# Chapter 7

### **Measurement of Particulates**

Part One PM<sub>2.5</sub> Monitoring

Part Two
PM<sub>10</sub> Monitoring

<u>Part Three</u> Total Suspended Particulate Monitoring for Metals

Part Four
Continuous PM<sub>10</sub> and PM<sub>2.5</sub> Monitoring

<u>Part Five</u> PM<sub>2.5</sub> Speciation Monitoring

## PART ONE

PM<sub>2.5</sub> Monitoring

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### Part One - PM<sub>2.5</sub> Monitoring

#### 1.0 Introduction

Between the years 1900 and 1970, the emission of six principal ambient air pollutants increased significantly. The principal pollutants, also called criteria pollutants, are: particulate matter  $(PM_{10} \& PM_{2.5})$ , sulfur dioxide  $(SO_2)$ , carbon monoxide (CO), nitrogen dioxide  $(NO_2)$ , ozone  $(O_3)$ , and lead (Pb). In 1970 the Clean Air Act (CAA) was signed into law. The CAA and its amendments provide the framework for all pertinent organizations to protect air quality. This framework provides for the monitoring of these criteria pollutants by State and local organizations through the Air Quality Monitoring Program.

The criteria pollutant defined as particulate matter is a general term used to describe a broad class of substances that exist as liquid or solid particles over a wide range of sizes. As part of its Ambient Air Quality Monitoring Program, the Indiana Department of Environmental Management, Office of Air Quality (IDEM, OAQ) will measure two particle size fractions; those less than or equal to 10 micrometers (PM<sub>10</sub>), and those less than or equal to 2.5 micrometers (PM<sub>2.5</sub>). Part One of Chapter 7 focuses on the activities associated with PM<sub>2.5</sub>.

The background and rationale for the implementation of Indiana's PM<sub>2.5</sub> ambient air monitoring network are found in the Federal Register. In general, some of the findings are listed below.

- The characteristics, sources, and potential health effects of larger or "coarse" particles (from 2.5 to 10 micrometers in diameter) and smaller or "fine" particles (smaller than 2.5 micrometers in diameter) are very different.
- Coarse particles come from sources such as windblown dust from agricultural fields and dust kicked up on unpaved roads from vehicle traffic.
- Fine particles are generally emitted from activities such as industrial and residential combustion and from vehicle exhaust. Fine particles are also formed in the atmosphere from gases such as sulfur dioxide, nitrogen oxides, and volatile organic compounds that are emitted from combustion activities and then become particles as a result of chemical transformations in the air.
- Coarse particles can deposit in the respiratory system and contribute to health effects such as
  aggravation of asthma. EPA's "staff paper" concludes that fine particles, which also deposit
  deeply in the lungs, are more likely than coarse particles to contribute to the health effects
  (i.e., premature mortality and hospital admissions) found in a number of recently published
  community epidemiological studies.

- These recent community studies find that adverse public health effects are associated with exposure to particles at levels well below the current standards for both short-term (i.e., less than 1 day to up to 5 days) and long-term (generally a year to several years) periods.
- Health effects include premature death and increased hospital admissions and emergency
  room visits (primarily among the elderly and individuals with cardiopulmonary disease);
  increased respiratory symptoms and disease (among children and individuals with
  cardiopulmonary disease such as asthma); decreased lung function (particularly in children
  and individuals with asthma); and alterations in lung tissue and structure and in respiratory
  tract defense mechanisms.

Air quality samples are generally collected for one or more of the following purposes:

- 1. To judge compliance with and/or progress made towards meeting the NAAQS
- 2. To develop, modify or activate control strategies that prevent or alleviate air pollution episodes
- 3. To observe pollution trends throughout the region, including non-urban areas
- 4. To provide a data base for research and evaluation of effects

With the end use of the air quality samples as a prime consideration, various networks can be designed to meet one of six basic monitoring objectives listed below:

- Determine the highest concentrations to occur in the area covered by the network.
- Determine representative concentrations in areas of high population density.
- Determine the impact on ambient pollution levels of significant source or source categories,
- Determine general background concentration levels.
- Determine the extent of Regional pollutant transport among populated areas, and in support of secondary standards.
- Determine the welfare-related impacts in more rural and remote areas.

In general, the measurement goal of the Indiana's  $PM_{2.5}$  Ambient Air Quality Monitoring Program is to estimate the concentration, in units of micrograms per cubic meter ( $\mu g/m^3$ ) of particulates less than or equal to 2.5 micrometers ( $\mu m$ ) that have been collected on a small filter for a period of 24 hours. The majority of Indiana's  $PM_{2.5}$  monitoring stations are a part of the SLAMS network. The primary goal of this network is to compare the  $PM_{2.5}$  concentrations to the annual and 24-hour National Ambient Air Quality Standard. The national primary and secondary ambient air quality standards for  $PM_{2.5}$  are 15.0 micrograms per cubic meter ( $\mu g/m^3$ ) annual arithmetic mean concentration and 65  $\mu g/m^3$  24-hour average concentration measured in ambient air.

The reference method for sampling PM<sub>2.5</sub> is found in 40 CFR Part 50, Appendix L and 40 CFR part 53, Subpart E. In general, the sampling involves drawing a measured quantity of ambient air at a constant volumetric flow rate through a specially designed particulate size selective inlet.

 $PM_{2.5}$  particles are those with an aerodynamic diameter of less than or equal to 2.5  $\mu$ m. Particles in the 2.5 $\mu$ m and smaller size range are collected on a 46.2-millimeter polytetrafluoreoethylene filter during the specified 23 to 25-hour sampling period. Each filter is weighed before and after sampling. From these measurements, the mass of the collected  $PM_{2.5}$  sample is calculated.

The total volume of air sampled is determined from the measured volumetric flow rate and the sampling time. The concentration of  $PM_{2.5}$  in the ambient air is computed as the total mass of collected particles in the  $PM_{2.5}$  size range divided by the total volume of air sampled and measured under the ambient (actual) conditions of temperature and pressure. The  $PM_{2.5}$  concentration is expressed as micrograms per cubic meter ( $\mu g/m^3$ ) of air.

### 2.0 Network Categorization/Description

Indiana's monitoring network consists of four major categories of monitoring stations that measure the criteria pollutants. The  $PM_{2.5}$  monitoring program falls into two of these categories, the State and Local Air Monitoring Stations (SLAMS), and Special Purpose Monitoring (SPM) categories. These stations are described below.

- SLAMS consist of a national network of ~ 3,500 monitoring stations whose size and distribution is largely determined by the needs of State and local air pollution control agencies to meet their respective State implementation plan (SIP) requirements.
- Special Purpose Monitoring Stations (SPMS) provide for special studies needed by the State and local agencies to support their State implementation plans (SIP's) and other air program activities. The SPMS are not permanently established and, thus, can be adjusted easily to accommodate changing needs and priorities. The SPMS are used to supplement the fixed monitoring network as circumstances require and resources permit. If data from SPMS is used for SIP purposes, SPMS must meet all QA and methodology requirements for SLAMS monitoring.

### 3.0 Field Activities

The performance requirements of the air sampler has been specified in 40 CFR Part 50, Appendix L of the 7/18/97 Federal Register Notice. Table 1 summarizes some of the critical performance requirements.

The IDEM, OAQ Air Toxics Section receives all PM<sub>2.5</sub> filters from the USEPA. Acceptance criteria testing for these filters have been performed by EPA (or its contractors); however, the Air Toxics Section's staff examines each filter for pinholes, tears and other defects.

The IDEM, OAQ Ambient Monitoring Section evaluates each newly received sampler using an acceptance/rejection checklist that includes the performance criteria listed in Table 1.

Table 2 summarizes the field measurements that must be collected by the sampler and sampler operator. This table is presented in 40 CFR Part 50, as Table L-1 of Appendix L. These measurements are made by the air sampler and are stored in the instrument for downloading by the field operator during routine sample set-up and sample pickup visits. Some sites are equipped with a modem for downloading via a telephone line.

Table 1
Performance / Design Specifications

Equipment	Frequency	Acceptance Criteria	Reference
Filter Design	Vendor Cert.	see reference	40 CFR Part 50, App. L, Sec 6.0
Specifications	44		
Size	44	46.2 mm dia ±0.25 mm	Sec 6.1
Medium	"	Polytetrafluoroethylene	Sec 6.2
Support ring		Polymethylpentene, 0.38 mm thick, 46.2 mm ±0.25 mm outer dia., 3.68 (±0.00, -0.51 mm) width	Sec 6.3
Pore size	44	2 μm	Sec 6.4
Filter thickness	"	30-50 μm	Sec 6.5
Max. pressure drop	"	30 cm H <sub>2</sub> O @ 16.67 l/min	Sec 6.6
Max. Moisture pickup	"	10 μg increase in 24 hr.	Sec 6.7
Collection efficiency	"	99.7%	Sec 6.8
Filter weight stability	"	<20 μg	Sec 6.9.1 and 6.9.2
Alkalinity	"	<25.0 microequivalents/gram	Sec 6.10
Sampler Performance Specifications	All Instruments		
Sample Flow Rate	"	1.000 m <sup>3</sup> /hr.	
Flow Regulation	"	$1.000 \pm 5\% \text{ m}^3/\text{hr}.$	40 CFR Part 50, App. L 50,
Flow Rate Precision	"	2% CV	Sec7.4
Flow Rate Accuracy	"	±2%	٠,
External Leakage	"	Vendor specs	
Internal Leakage	"	Vendor specs	٠٤
Ambient Temp Sensor	"	-30° - 45 °C	
T	44	1 °C res. ±1.6 °C accuracy	
Filter Temp Sensor	••	-30° - 45 °C	Vol-II -MS. 2.12
Danamatria Danama	"	0.1 °C res. ±1.0 °C accuracy	40 CFR Part 50, App. L, Sec7.4
Barometric Pressure		600-800 mmHg	
Clock/Timer	"	5 mm res. ±10 mm accuracy Date/time.	٠.
CIOCK/ I IIIIEI		1 sec. res. ±1 min/month	
		accuracy	"

# Table 2 Field Measurements

	Appendix		Avai	ilability		For	mat
Information to be provided	L section reference	Any Time <sup>a</sup>	End of period <sup>b</sup>	Visual display <sup>c</sup>	Data output <sup>d</sup>	Digital reading <sup>e</sup>	Units
Flow rate, 30-second maximum interval	7.4.5.1	1	-	1	*	XX.X	l/min
Flow rate, average for the sample period	7.45.2	*	✓	*	1	XX.X	l/min
Flow rate, CV, for the sample period	7.4.5.2	*	✓	*	<b>√</b> ●	XX.X	%
Flow rate, 5-min average out of spec. (FLAG)	7.4.5.2	1	✓	1	∕•	On/Off	
Sample volume, total	7.4.5.2	*	✓	1	/●	XX.X	m <sup>3</sup>
Temperature, ambient, 30-second interval	7.4.8	1	-	1	-	XX.X	°C
Temperature, ambient, min., max., average for the sample period	7.4.8	*	✓	1	∕•	XX.X	°C
Barometric pressure, ambient, 30-second interval	7.4.9	1	-	1	-	XXX	mmHg
Barometric pressure, ambient, min., max., average for the sample period	7.4.9	*	<b>√</b>	1	✓•	XXX	mmHg
Filter temperature, 30-second interval	7.4.11	1		1		XX.X	°C
Filter temperature, differential, 30-minute interval, out of spec. (FLAG)f	7.4.11	*	✓	1	<b>√</b> ●	On/Off	
Filter temperature, maximum differential from ambient, date, time of occurrence	7.4.11	*	*	**	*	X.X, YY/MM/DD HH:mm	°C, Yr/Mo/ Day Hr min
Date and time	7.4.12	1	-	1	-	YY/MM/DD HH:mm	Yr/Mo/ Day Hr min
Sample start and stop time settings	7.4.12	1	✓	1	1	YY/MM/DD HH:mm	Yr/Mo/ Day Hr min
Sample period start time	7.4.12	-	✓	1	∕•	YYYY/MM DD HH:mm	Yr/Mo/ Day Hr min
Elapsed sample time	7.4.13	*	✓	1	/●	HH:mm	Hr min
Elapsed sample time out of spec. (FLAG) <sup>f</sup>	7.4.13	-	✓	1	∕•	On/Off	
Power interruptions >1 min, start time of first 10	7.4.15.5	*	✓	*	1	1HH:mm, 2HH:mm, etc.	Hr min
User-entered information, such as sampler and site identification	7.4.16	1	✓	1	<b>√●</b>	As entered	

 <sup>✓</sup> Provision of this information is required.

\*\*Provision of this information is optional. If information related to entire sample period is optionally provided prior to end of sample period, value provided should be value calculated for the portion of sampler period completed up to the time information is provided.

<sup>•</sup> Indicates that this information is also required to be provided to the AQS data bank.

<sup>&</sup>lt;sup>a</sup> Information is required to be available to the operator at any time the sampler is operating, whether sampling or not.

<sup>&</sup>lt;sup>b</sup> Information relates to the entire sampler period and must be provided following the end of the sample period until reset manually by the operator or automatically by the sampler upon the start of a new sample period.

<sup>&</sup>lt;sup>c</sup> Information shall be available to the operator visually.

<sup>&</sup>lt;sup>d</sup> Information is to be available as digital data at the sampler's data output port following the end of the sample period until reset manually by the operator or automatically by the sampler upon the start of a new sample period.

e Digital readings, both visual and data output, shall have no less than the number of significant digits and resolution specified.

<sup>&</sup>lt;sup>f</sup> Flag warnings may be displayed to the operator by a single-flag indicator or each flag may be displayed individually. Only a set (on) flag warning must be indicated; an off (unset) flag may be indicated by the absence of a flag warning. Sampler users should refer to Section 10.12 of Appendix L regarding the validity of samples for which the sampler provided an associated flag warning.

### 4.0 Sampler Components

Each PM<sub>2.5</sub> sampler used by IDEM, OAQ to report data to the AQS or for use in determining Indiana's attainment of the National Ambient Air Quality Standards (NAAQS) must meet the requirements set forth in 40 CFR Parts 50 and 53.

In general the components for a PM<sub>2.5</sub> sampler include:

- Sample air inlet
- Down tube
- Particle size separator (impactor),
- Impaction jet, Impaction filter, Impaction oil
- Filter holder assembly, Upper Portion, Lower Portion
- Air Pump
- Flow rate control system: capable of providing a constant design flow rate of 16.67 l/min to the sampler inlet at ambient conditions
- Flow rate measurement device capable of measuring flow rate at the sampler inlet within ±2 percent: available for display at anytime, updated every 30 seconds
- Ambient and filter temperature monitoring system
- Ambient barometric pressure monitoring system
- Timer: capable of measuring time to an accuracy of  $\pm$  minute/month, capable of measuring elapsed time to within  $\pm$  minute/1440 minutes
- Electrical system capable of meeting or exceeding design specifications

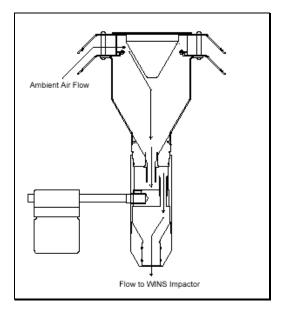


Figure 1 PM<sub>2.5</sub> Sample Inlet

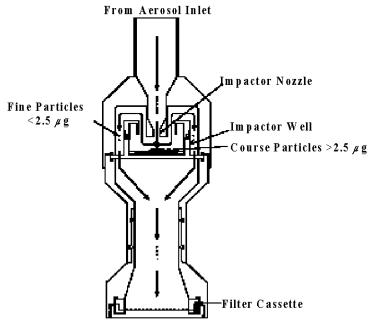


Figure 2 PM<sub>2.5</sub> WINS Impactor

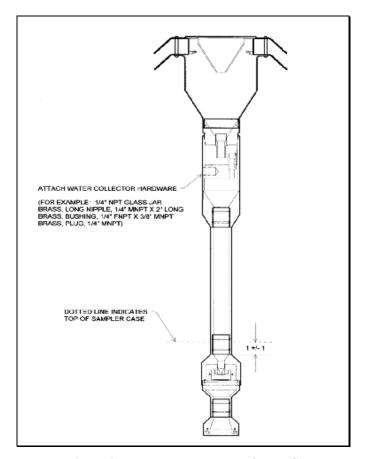


Figure 3 PM<sub>2.5</sub> Inlet, Downtube & WINS

### 5.0 Sampler Installation

### **5.1** Siting Requirements

As with any type of air monitoring study in which the sample data are to be used to draw conclusions about a geographic area, the validity of those conclusions depends on the representativeness of the sampling data. Therefore, an initial goal of the Indiana's  $PM_{2.5}$  monitoring project is to select a site where the  $PM_{2.5}$  measurements are representative of the monitoring area.

Spatial and temporal scale considerations are important in PM<sub>2.5</sub> sampler siting. Spatial scales may range from a small (0.1 - to 0.5 km<sup>2</sup>) area to large regional areas exceeding tens of hundreds of square kilometers. Whether the potential impact of particulate pollution is generated by a local or general source category will affect the decision on the size of the spacial monitoring scale. In addition, the siting of the samplers within the monitoring network should reflect whether the expected impact is limited to a small area (a few city blocks) or will extend to larger areas (metropolitan and rural). With regard to the temporal scale, interest focuses on either the annual or the geometric mean concentration or a 24-hour average concentration. Because siting of a PM<sub>2.5</sub> sampler requires considering the prevailing wind direction, a sampler sited for monitoring trends in air quality over a period of a year will not necessarily be ideal for measuring 24-hour concentrations. Thus, the choice of temporal aspects of the network design and optimum exposure are more completely explained in 40 CFR Part 50, Appendix L and Part 58, Appendix D and in the siting guidelines outlined in Chapter 1 Section 4.0, of the Indiana Department of Environmental Management, Office of Air Quality's Quality Assurance Manual.

Although spatial and temporal scales must be considered in site selection, the following guidelines are observed by IDEM, OAQ regardless of scale:

- The PM<sub>2.5</sub> sampler has an unobstructed airflow for a minimum of 2 m in all directions.
- The sampler inlet is placed at a height of 2 to 15 m above ground level.
- When a PM<sub>2.5</sub> sampler is collocated with any other particulate matter sampler, the spacing between the sampler inlets is at least 1 m and no more than 4 m. The heights of the inlets should be within 1m as measured in a vertical direction.

The IDEM, OAQ also considers the following additional factors when determining sampler location at a site. These factors include:

Accessibility under all weather conditions
 All IDEM, OAQ PM<sub>2.5</sub> samplers used for routine sampling are situated where the operator
 can reach it safely regardless of weather conditions. Samplers located on rooftops are placed
 so that an operator's personal safety is not jeopardized by a slippery roof surface during
 inclement weather. Considerations are also given to the fact that routine operations (i.e.,
 calibrations, sampler filter installation and recovery, flow checks and audits) involve
 transporting equipment and supplies to and from the monitoring site.

- Availability of adequate electricity 40 CFR Part 50, Appendix L, specifies that a PM<sub>2.5</sub> sampler is required to operate at 105-125 volts, AC and at a frequency of 59-61 Hz. The sampler may pull a higher current when the pump starts, possibly necessitating a slow-blow fuse. Although PM<sub>2.5</sub> samplers are required to indicate power interruptions, every effort is made to provide a stable source for the monitoring site.
- Security of monitoring personnel and equipment
   The security of personal and the sampler itself depends largely on location. The IDEM, OAQ utilizes rooftop sites with locked access and ground level sites with fences whenever possible.

### **5.2** Sampler Installation Procedures

### 5.2.1 Receipt of Sampler

On receipt of any new equipment from the manufacturer the IDEM, OAQ, Ambient Monitoring Section (AMS) personnel follow a general checklist:

- ✓ Unpack and inspect each sampler to insure that all components are present.
- ✓ Compare equipment delivered with items enclosed on the packing slip. The IDEM Distribution Center (formerly Stores and Mails) and the manufacturer are notified immediately of any missing or damaged equipment.
- ✓ Read the manufacturer's instruction manual and become familiar with the sampler's operating and calibration procedures.
- ✓ Assemble the sampler in the AMS laboratory according to the manufacturer's instructions.
- ✓ Develop equipment specific SOPs and train staff in operation and maintenance.

On receipt of each new  $PM_{2.5}$  sampler from the manufacturer, the IDEM, OAQ, AMS personnel run through the following general check list of testing and acceptance criteria:

- ✓ Check the enclosed packing list. Were all parts listed included in the delivery of the monitor? Yes / No
- ✓ Were any of the enclosed parts broken during the shipping of the monitor? Yes / No
- ✓ Check the enclosed assembly instructions. Did all parts fit together during the assembly of the monitor? Yes / No

- ✓ Does the monitor turn on when supplied with electrical power? Yes / No
- ✓ Check to see if the timer will automatically turn on and off during a set time by setting the timer to start and stop the monitor while the operator is present. Yes / No
- ✓ Does the computer boot up and operate properly? Check to see if the computer has working software by performing manual input of information into the computer. Yes / No
- ✓ Does the computer download information properly? Check this by manually trying to download information. Yes / No
- ✓ Does the internal fan operate properly? Check this by supplying electrical power to the unit and checking if the fan will turn on and off. Yes / No
- ✓ Does the filter holder apparatus operate properly? Check this by manually installing a filter into the holder apparatus and checking to see if the filter is sealed into the unit. Yes / No
- ✓ Does the casing protect the internal unit from weather? Check this by visually inspecting the unit's gaskets and seals for holes, leaks, etc. Yes / No
- ✓ Does the unit support structure keep the unit secure and upright? Yes / No
- ✓ When all parts are assembled and operated together, does the unit function properly? Check this by assembling the unit as the instructions dictate, installing a filter, setting the timer, and operating the unit as a normal monitoring period. Yes / No

(Accept / Reject)

### **5.2.2 Laboratory Evaluation**

Upon the acceptance of the new sampler from the manufacturer, the IDEM, OAQ, Ambient Monitoring Section (AMS) personnel perform the following general activities:

- Turn-on the sampler.
- Perform a leak check according to the manufacturer's instructions.
- Calibrate the flow rate at 16.7 l/min according to the manufacturer's instructions.
- Perform audits of the temperature and pressure sensors according to the manufacturer's instructions.
- Perform a flow check according to the manufacturer's instructions.

### 5.2.3 Set-up at the Sampling Site

Prior to sampler set-up at the site and to simplify the sampler installation, all Indiana  $PM_{2.5}$  sites have been equipped with 4 feet by 4 feet platforms. Platforms are designed with mounting bolts and nuts to facilitate the easy installation of each sampler.

### **5.2.4 Field Evaluation**

- Secure the sampler onto a platform.
- Plug the power cord into the voltage outlet and energize the sampler. Electrical connections should be waterproofed to ensure operator safety and to avoid short-circuit and/or power interruptions. All electrical connections must be installed so as not to become submerged in water during periods of inclement weather.
- Perform calibrations of the temperature and pressure sensors.
- Perform flow calibrations according to the manufacturer's instructions.
- Install a WINS impactor in the sampler.
- Program sample frequency, start date.
- Load sampler with filter magazines.

The sampler is now ready for routine sample collection.

### 6.0 Project/Task Description

### 6.1 Description of Work to be Performed

In general, the measurement goal of the  $PM_{2.5}$  Ambient Air Quality Monitoring Program is to estimate the concentration, in units of micrograms per cubic meter ( $\mu g/m^3$ ), of particulates less than or equal to 2.5 micrometers ( $\mu m$ ). For Indiana's monitoring network, the primary goal is to compare the  $PM_{2.5}$  concentrations to the annual and 24-hour National Ambient Air Quality Standard (NAAQS). The national primary and secondary ambient air quality standards for  $PM_{2.5}$  are 15.0 micrograms per cubic meter ( $\mu g/m^3$ ) annual arithmetic mean concentration and 65  $\mu g/m^3$  24-hour average concentration measured in ambient air. A description of the NAAQS and its calculation can be found in the 1997 Federal Register Notice. In addition, Appendix L of part 50 also provides the following summary of the measurement principle:

An electrically powered air sampler draws ambient air at a constant volumetric flow rate into a specially shaped inlet and through an inertial particle size separator (impactor) where the suspended particulate matter in the  $PM_{2.5}$  size range is separated for collection on a polytetrafluoroethylene (PTFE) 46.2 mm filter over the specified sampling period.

Each filter is weighed (after moisture and temperature equilibration) before and after sample collection to determine the net weight (mass) gain due to collected  $PM_{2.5}$ . The total volume of air sampled is determined by the sampler from the measured flow rate at actual ambient temperature and pressure and the sampling time. The mass concentration of  $PM_{2.5}$  in the

ambient air is computed as the total mass of collected particles in the PM<sub>2.5</sub> size range divided by the actual volume of air sampled, and is expressed in micrograms per actual cubic meter of air  $(\mu g/m^3)$ .

The following sections describe the measurements required for the routine field and laboratory activities for the network.



Photo 1 R&P 2025 Sampler: Kokomo Fire Station

### **6.2** Field Activities

Design and performance requirements of PM<sub>2.5</sub> air samplers are specified in Part 50, Appendix L of the 7/18/97, Federal Register Notice. Table 1 summarizes these requirements.

The R&P Partisol-Plus Air Samplers have been purchased, distributed, and certified by the EPA as meeting the requirements specified in the Federal Register. Therefore, Indiana assumes the sampling instruments to be adequate for the sampling for  $PM_{2.5}$ .

### **6.2.1 Field Measurements**

Table 2 and Table 3 list field measurements that must be collected. These measurements are made by the R&P Partisol-Plus Air Sampler and are stored in the instrument for downloading by the field operator during routine visits.

# Table 3 Additional Field Measurements

Parameter	Example Parameter Code	Frequency	Units	Comment
Monitor ID	MONID	Every sample event	see AQS	Unique AQS Monitor ID that include the combination of STATE, COUNTY, SITE, PARAMETER, and POC fields
Site Name	SITENAM	Every sample event	AAA	Unique site name associated with the site
Sampler ID	SAMPID	Every sample event	AAXXX	Sampler model number or unique bar code number associated with the model number
QC Thermometer ID Initial	QCTIDI	Every sample event	AAAXXX	Unique ID number of QC thermometer used for ambient air temp check at the beginning of sampling
QC Temperature Measurement Initial	QCTEMPI	Every sample event	XX°C	QC temp reading at the beginning of sampling
QC Baromtr ID Initial	QCBIDI	Every sample event	AAAXXX	Unique alpha-numeric ID of QC barometric pressure device used for barometric pressure reading check
QC Bar. Pressure Reading Initial	QCBI	Every sample event	XXX mmHg	QC temp reading at sample start
QC Thermometer ID Final	QCTIDF	Every sample event	AAAXXX	Unique ID number of QC thermometer used for ambient air temp check at the beginning of sampling
QC Temperature Measurement Final	QCTEMPF	Every sample event	XX°C	QC temp reading at the end of sampling
QC Baro. ID Final	QCBIDF	Every sample event	AAAXXX	Unique alpha-numeric ID of QC barometric pressure device used for barometric pressure reading check
QC Bar. Pressure Reading Final	QCBF	Every sample event	XXX mmHg	QC temp reading at the end of sampling
Filter ID	FID	Every sample event	AAYYXXXX	Unique filter ID of filter given by the weighing laboratory.
Filter Integrity flag	FFIF	Every sample event	QFI/ VFI/GFI	QFI -Questionable filter integrity VFI- Void Filter Integrity GFI-Good Filter Integrity
Site Operator Initial	SOI	Every sample event	AAA	Initials of the site operator setting up the sampling run
Site Operator Final	SOF	Every sample event	AAA	Initials of the site operator completing the sampling run
Free Form Notes	FFM	As needed	AAA	Free form notes about the sampling run

### **6.3** Laboratory Activities

PM<sub>2.5</sub> laboratory activities include preparing filters for routine field operations.

- 1. Pre-Sampling weighing
  - filter receipt from EPA
  - check filter integrity
  - condition filter
  - weigh filters
  - store prior to field use
  - packaging filters for field use
  - QA/QC activities
  - maintain microbalance at specified environmental conditions
  - equipment maintenance and calibrations

### 2. Shipping/Receiving

- filter receipt and log-in from the field
- filter storage
- QA/QC activities

### 3. Post-Sampling Weighing

- check filter integrity
- weigh filters
- data download from field data loggers
- data entry/upload to AQS
- store filters/archiving
- QA/QC activities

Details for these activities are included in various sections of this document as well as *Guidance Document 2.12*. Table 4 provides the performance specifications of the laboratory environment and equipment.

Table 4
Laboratory Performance Specifications

Equipment	Acceptance Criteria
Microbalance	Resolution of 1 µg, repeatability of 1 µg
Microbalance environment	Climate-controlled, draft-free room or chamber or equivalent. Mean relative humidity 30 to 40%, with a variability of not more than $\pm 5\%$ over 24 hours. Mean temperature should be held between 20 to 23 °C, with a variability of not more than $\pm 2$ °C over 24 hours.
Mass reference standards	Standards bracket weight of filter, individual standard's tolerance less than 25 µg, handle with smooth, nonmetallic forceps

# **6.3.1** Laboratory Measurements

Table 5 lists the parameters that are recorded for pre- and post-sampling laboratory activities.

Table 5
Laboratory Measurements

Parameter	Example Parameter Code	Frequency	Units	Comments
Conditioning Start Date	CNSDATE	every filter	YY/MM/DD	Date of start of conditioning period
Start Time	CNSHOUR	every filter	XX.XX	Start hour and minute of conditioning
Filter Number (No.)	RFID, LBFID, FBID	every filter	RFYYXXXX LBYYXXXX FBYYXXXX	Unique filter ID of routine filter (RF) Lab Blanks (LB) Field Blanks (FB)
Relative Humidity	CONRH	1/run	XX%	Avg % relative humidity for conditioning session based upon readings every 10 minutes
Temperature	CONTEMP	1/run	XX°C	Average temperature value for conditioning session based upon readings every 10 minutes
End Date	CONDATE	every filter	YY/MM/DD	Date of start of conditioning period
End Time	CNEHOUR	every filter	XX.XX	End hour and minute of conditioning
Pre-Sampling Filter Weighing Date	PREDATE	1/run	YY/MM/DD	Date for pre-sampling run of filters associated with each filter
Lot Number	FLN	every filter	AAAXXX	Lot number associated with filter
Balance No.	BALID	1/run	AAAXXX	Balance ID for balance used in pre-weighing
Analyst	PREANL	1/run	AAA	Initials of the technician preweighing filters
QA Officer	PREQC	1/run	AAA	Initials of the QA Officer overseeing preweighing filters
Relative Humidity	PRERH	1/run	XX%	Average % relative humidity value for weighing session based upon readings every 10 min.
Temperature	PRETEMP	1/run	XX°C	Average temperature value for weighing session based upon readings every 10 min.
Filter Number	RFID LBFID FBID FCID DFID	every filter	RFYYXXX LBYYXXXX FBYYXXXX FCYYXXXX DFYYXXXX	Unique filter ID of routine filter (RF) Lab Blanks (LB) Field Blanks (FB) Flow Check Filter (FC) and Duplicate Filter
QC Sample Number	PREQC	every QC check	C1XXX C2XXX C3XXX	Unique ID for calibration checks and or other types of QC samples used
Pre-Sampling Mass	PREMASS	every filter	XXX.XXX mg	Mass weight in mg of the filter
Transport container ID	CONTID	every filter	AAAXXX	Identification of the filter transport container

Parameter	Example Parameter Code	Frequency	Units	Comments
Monitor ID	MONID	Every sample	see AQS	AQS Monitor ID: STATE, COUNTY, SITE, PARAMETER, & POC fields
Free Form Notes	PREFFM	As needed		Pre-weighing Free Form notes
Post-Sampling Filter Weighing Date	PSTDATE	1/run	YY/MM/DD	Date for post-sampling run of filters that can then be associated with each filter
Balance Number	BALID	1/run	AAAXXX	Unique balance ID for balance used in post- weighing
Analyst	PSTANL	1/run	AAA	Initials of the technician post-weighing filters
QA Officer	PSTQC	1/run	AAA	Initials of the QA Officer overseeing preweighing filters
Relative Humidity	PSTRH	1/run	XX%	Average % relative humidity value for weighing period based upon readings every 10 minutes
Temperature	PSTEMP	1/run	XX°C	Average temperature value for weighing period based upon readings every 10 minutes
Filter Number	RFID LBID FBID DFID	every filter	RFYYXXX LBYYXXXX FBYYXXXX DFYYXXXX	Unique filter ID of routine filter (RF) Lab Blanks (LB) Field Blanks (FB) and Duplicate Sample
QC Sample Number	PSTQC	every QC check	C1XXX C2XXX C3XXX	Unique id for calibration checks and or other types of QC samples used
Post Sampling Mass	PSTMASS	every filter	XXX.XXX mg	Mass weight in mg of the filter
Net Mass	NETMASS	every filter	XX.XXX mg	Net weight (PSTMASS-PREMASS)-in mg of PM <sub>2.5</sub> catch.
Weighing Flag	PSTFLAG	as needed	AAA	Flags associated with concentration
Free Form Notes	PSTFFM	as needed	AAA	Post weighing free form notes

## **6.4** Project Assessment Techniques

An assessment is an evaluation process used to measure the performance or effectiveness of a system and its elements. As used here, assessment is an all-inclusive term used to denote any of the following: audit, performance evaluation (PE), management systems review (MSR), peer review, inspection, or surveillance.

Table 6 Assessment Schedule

Assessment Type	Assessment Agency	Frequency
Technical Systems Audit	EPA Region 5	1 every 3 years
Technical Systems Audit	IDEM QA Section	1 every 3 years
Network Review	EPA Region 5	every year
Network Review	IDEM Air Monitoring Branch	App D 1/year, App E 1/3 years
FRM Performance Evaluation	EPA Region 5	25% of sites/year/4 times per year
Data Quality Assessment	IDEM Air Monitoring Branch	every year

# 6.5 Schedule of Activities

 $\label{eq:Table 7} Table \ 7$  Schedule of Critical PM  $_{2.5}$  Activities (Plan, implement and assess the program)

Activity	Due Date	Comments
Network development	Jan 15, 1998	Preliminary list of sites and samplers required
Sampler order	Mar 2, 1998	Samplers ordered from National contract
Laboratory design	Feb 1, 1998	Listing of laboratory requirements
Laboratory procurement	April 1, 1998	Ordering/purchase of all laboratory & misc. field equipment
Personnel Requirements	April 1, 1998	Advertising for field and laboratory personnel (if required)
QAPP development	May-Nov., 1998	Development of the QAPP
Network design completion	Jul 1, 1998	Final network design
Samplers arrive	Jul 1, 1998	Arrival of FRM samplers
Sampler siting/testing	Jul-Dec, 1998	Establishment of sites and preliminary testing of samplers
Field/Laboratory Training	Aug, 1998	Field and laboratory training activities and certification
QAPP Submittal	Dec 15, 1998	QAPP Submittal to EPA
QAPP Approval	Jan 26, 1999	Approval by EPA
Pilot testing	Aug-Dec 1998	Pilot activities to ensure efficiency of measurement system
Installation of 1998 sites	Dec 31, 1998	Sites must be established and ready to collect data
Routine Sampling	Jan 1, 1999	Routine activities must start

### 6.6 Project Records

The IDEM had established standard operating procedures (SOPs) for the timely preparation, review, approval, issuance, use, control, revision, and maintenance of documents and records. Table 8 represents the categories and types of records and documents which are applicable to document control for  $PM_{2.5}$  information. Information on key documents in each category is explained in detail in Section 9.

Table 8
Critical Documents and Records

Categories	Record/Document Types	
	State Implementation Plan	
	Reporting agency information	
	Organizational structure	
Manager 1 Octobries	Personnel qualifications and training	
Management and Organization	Training Certification	
	Quality management plan	
	Document control plan	
	EPA Directives, Grant allocations, Support Contract	
	Network description	
Site Information	Site characterization file	
	Site maps, Site Pictures	
	QA Project Plans	
	Standard operating procedures (SOPs)	
Environmental Data Operations	Field and laboratory notebooks	
-	Sample handling/custody records	
	Inspection/maintenance records	
Raw Data	Any original data (routine and QC data) including data entry forms	
	Air quality index report	
Data Barantina	Annual SLAMS air quality information	
Data Reporting	Data/summary reports	
	Journal articles/papers/presentations	
	Data algorithms	
Data Managament	Data management plans	
Data Management	PM <sub>2.5</sub> Data	
	Data Management Systems	
	Good Laboratory Practice	
	Network reviews	
	Data quality assessments	
Quality Assurance	QA reports	
	System audits	
	Response/Corrective action reports	
	Site Audits	

### 7.0 Quality Objectives and Criteria for Measurement Data

### 7.1 Data Quality Objectives (DQOs)

DQOs are qualitative and quantitative statements derived from the DQO Process that clarify the monitoring objectives, define the appropriate type of data, and specify the tolerable levels of decision errors for the monitoring program. By applying the DQO Process to the development of a quality system for PM<sub>2.5</sub>, the EPA guards against committing resources to data collection efforts that do not support a defensible decision. During the months from April to July of 1997, the DQO Process was implemented by EPA for the PM<sub>2.5</sub>. The DQOs were based on the data requirements of the decision maker(s). Regarding the quality of the PM<sub>2.5</sub> measurement system, the objective is to control precision and bias in order to reduce the probability of decision errors.

### 7.2 Measurement Quality Objectives (MQOs)

Once a DQO is established, the quality of the data must be evaluated and controlled to ensure that it is maintained within the established acceptance criteria. Measurement quality objectives are designed to evaluate and control various phases (sampling, preparation and analysis) of the measurement process to ensure that total measurement uncertainty is within the range prescribed by the DQOs. MQOs can be defined in terms of the following data quality indicators:

<u>Precision</u> - A measure of mutual agreement among individual measurements of the same property usually under prescribed similar conditions. This is the random component of error. Precision is estimated by various statistical techniques using some derivation of the standard deviation.

<u>Bias</u> - The systematic or persistent distortion of a measurement process which causes error in one direction. Bias is determined by estimating the positive and negative deviation from the true value as a percentage of the true value.

<u>Representativeness</u> - A measure of the degree which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.

<u>Detectability</u> - The determination of the low range critical value of a characteristic that a method specific procedure can reliably discern.

<u>Completeness</u> - A measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct, normal conditions. Data completeness requirements are included in the reference methods (40 CFR Part 50).

Comparability - A measure of confidence with which one data set can be compared to another.

Accuracy has been a term frequently used to represent closeness to "truth" and includes a

combination of precision and bias error components. This term has been used throughout the CFR and in some of the sections of this document. If possible, the Department will attempt to distinguish measurement uncertainties into precision and bias components.

For each of these attributes, acceptance criteria can be developed for various phases of the EDO (Environmental Data Operation). Various parts of 40 CFR have identified acceptance criteria for some of these attributes as well as *Guidance Document 2.12*. In theory, if these MQOs are met, measurement uncertainty should be controlled to the levels required by the DQO. Table 9 lists the MQOs for PM<sub>2.5</sub> program. More detailed descriptions of these MQO's and how they are used to control and assess measurement uncertainty is described in other elements, as well as SOPs of this document.

Table 9
Measurement Quality Objectives - Parameter PM<sub>2.5</sub>

Requirement	Frequency	Acceptance Criteria	40 CFR Reference	QA 2.12 Reference
Filter Holding Times Presampling	all filters	<30 days before sampling	Part 50, App.L, Sec 8.3	Section 7.8
Post-sampling weighing	"	<10 days at 25 °C <30 days at 4 °C	"	Section 7.10
Reporting Units	All data	μg/m <sup>3</sup>	Part 50.3	Section 11.1
Detection Limit Lower DL Upper Conc. Limit	All data All data	2 μg <sup>3</sup> 200 μg <sup>3</sup>	Part 50, App L, Sec 3.1 Part 50, App L, Sec 3.2	
Data Completeness	quarterly	75%	Part 50, App N, Sec 2.1	
Filter Visual defect check Filter Condition. Env.	All filters	See reference	Part 50, App. L Sec 6.0	Section 7.5
Equilibration Temp. Range	All filters	24 hrs. minimum 20-23 °C	Part 50, App. L Sec 8.2	Section 7.6
Temp. Control Humidity Range	"	±2 °C over 24 hr ±5% RH over 24 hr.	"	"
Lot Blanks	3 filters per lot	less than 15 μg		Section 7.6

-				
Lab QC Checks Field Filter Blank	see 2.12 ref.	±30 μg change between weighings	Part 50, App L, Sec 8.2	Section 7.7
Lab Filter Blank	3 per weighing sessions	±15 μg change between weighings	"	,,
Balance Check	Beginning, every 10 <sup>th</sup> samples, end	≤3 µg		Section 7.9
Duplicate Filter Weighings	1 per sample batch	±15 μg change between weighings		Section 7.7
Calibration/verification				
Flow Rate (FR) Cal.	If multi-point failure	±2% of transfer standard	Part 50, App. L Sec 9.2	Section 6.3 & 6.6
FR multi-point verification	1/year	±2% of transfer standard	Part 50, App. L Sec 9.2.5	Section 8.3
One point FR verification	1/month	±4% of transfer standard	Part 50, App. L Sec 7.4	Section 8.3
External Leak Check	every 5 samples	80 ml/min	Part 50, App. L Sec 7.4	Section 8.3
Internal Leak Check	every 5 samples	80 ml/min	Part 50, App. L Sec 9.3	Section 8.3
Temperature Calibration	If multi-pt failure	±2% of standard	Part 50, App. L Sec 9.3	Section 6.4
Temp multi-point Verify.	on install, then 1/yr	±2 °C of standard	Part 50, App. L Sec 9.3	Section 6.4 and 8.2
One-point temp	1/month	±4 °C of standard	Part 50, App. L Sec 9.3	Section 6.4 and 8.2
Pressure Calibration	on installation, then 1/yr	±10 mmHg	Part 50, App. L Sec 9.3	Section 6.5
Pressure Verification	1/4 weeks	±10 mmHg	Part 50, App. L Sec 7.4	Section 8.2
Clock/timer Verification	1/4 weeks	1 min/mo.	1 a.cc o, 1 app. 2 occ 71.	Not described
Accuracy	25% 6 : 4/	.100	D . 50 4 4 G . 25	G .: 10.2
FRM Performance Eval.	25% of sites 4/yr	±10% ±4% of audit standard	Part 58 App. A Sec. 3.5	Section 10.3
Flow rate audit	½ wk automated	±4% of audit standard	Part 58 App. A Sec. 3.5	Section 10.2
External Lact- Charle	4/year manual	490 m1/min	Not described	Continue 10.2
External Leak Check Internal Leak Check	4/year	<80 ml/min <80 ml/min	Not described Not described	Section 10.2 Section 10.2
	4/year 4/year	<80 mi/min ±2 °C	Not described  Not described	Section 10.2 Section 10.2
Temperature audit Pressure audit	4/year 4/year	$\pm 10 \text{ mmHg}$	Not described Not described	Section 10.2 Section 10.2
Balance audit	1/year	manufacturer's specs	Not described  Not described	Section 10.2 Section 10.2
Precision	11 year	manuracturer 8 specs	TYOU GESCHIDEG	Section 10.2
Collocated samples	every 6 days for	CV < 10%	Part 58, App. A Sec 3.5	Section 10.3
Conocated samples	15% of sites	C V \ 10 /0	and 5.5	Section 10.5
Single analyzer	1/3 month	CV < 10%	Not described	Not described
Single analyzer Single analyzer	1/year	CV < 10%	Not described	Not described
Reporting Org.	1/3 month	CV < 10%	Not described	Not described
reporting org.	1,5 111011111	0. 10/0	1101 000011000	1101 000011000

Calibration & Check Standards				
~	1/	1207 - CNICT 4 11 41	D. 4 50 A I. C 0.1	0
Flow rate transfer std	1/year	±2% of NIST-traceable std.	Part 50 App L. Sec. 9.1 and 9.2	Section 6.3
Field thermometer	1/year	±0.1 °C resolution	Not described	Sec. 4.2, 8.3
		±0.5 °C accuracy		
Field barometer	1/year	±1 mmHg resolution	Not described	Sec. 4.2, 8.3
		±5 mmHg accuracy		
Working mass standards	3-6 months	0.025 mg	Not described	Sec. 4.3, 7.3
Primary mass standards	1/year	0.025 mg	Not described	Sec. 4.3, 7.3

### 8.0 Special Training Requirements

All personnel assigned to Indiana's  $PM_{2.5}$  Ambient Air Quality Monitoring Program meet the criteria (work experience, education, and related training) required by the Indiana Department of Environmental Management for each specific job position.

The Air Monitoring Branch maintains a database to track additional internal/external job related training and education. This database, as well as all records on personnel qualifications, is available for review during audit activities.

A list of the training courses offered through the Department of Environmental Management is available upon request from:

Office of Air Quality Operations Branch Mail Code: 61-50 100 North Senate Avenue Indianapolis, IN 46206-6015 IDEM staff members who are assigned duties in the PM<sub>2.5</sub> monitoring program attended the following training:

- ♦ The Air Pollution Training Institute (APTI) Satellite broadcast of the EPA PM <sub>2.5</sub> Training TO19-98 series. The series has included: Monitoring, Analytical and QA/QC.
- A 2-day seminar on the R&P Partisol-Plus Air Sampler, providing training in the areas of:
  - Routine preventive maintenance (o-ring inspection/replacement, WINS maintenance, etc)
  - Calibration and verification of temperature, pressure and flow rate sensors
  - Filter set-up/exchange
  - Filter transport requirements
  - Field blanks
- ◆ A one-day seminar held with members of the Illinois EPA and the Cook County Department of Environmental Control
- ♦ The EPA Region 5 PM<sub>2.5</sub> Operator 2-day Training Course. Providing instruction on:
  - PM<sub>2.5</sub> general monitoring requirements
  - FRM sampler operations
  - FRM sampler quality assurance
  - ESAT contractors
  - Sampling and laboratory Operations
  - Hands on training with the R&P Partisol-Plus Air Samplers that are being used in Indiana's PM<sub>2.5</sub> Air Quality Monitoring Program
- ♦ The Air and Waste Management Workshop for PM<sub>2.5</sub> Monitoring

### 9.0 Documentation and Records Management

The IDEM PM<sub>2.5</sub> Ambient Air Quality Monitoring Program requires the maintenance and retention of various documents and records. The IDEM is following the guidelines outlined in Section 14 of the *EPA Quality Assurance for Air Pollution Measurements Systems, Volume II, Ambient Air Specific Methods, Part I,* on acquisition and information management. Although this emphasizes automated data acquisition, much of the discussion is also applicable to manual methods. There are several reasons for maintaining complete, orderly records and properly managing data. Records will:

- provide information on mechanical problems that occur and document how the problems were corrected
- provide a history of warranty repairs
- provide a history of in-house repairs and preventive maintenance servicing
- document and date site placement details for the primary and collocated samplers as well as the characteristics of the surrounding land areas, sources, and other feature

- be a useful source of information at the time of the annual network review to show the proper sampler installation and operation, performance of QA/QC checks, traceability of equipment and standards, and proof that all systems were kept in control (use of control charts)
- provide evidence to support the quality of PM<sub>2.5</sub> data submitted to regional and national databases

### 9.1 Routine Data Activities

The IDEM has a structured records management retrieval system that allows for the efficient archival and retrieval of records. The PM<sub>2.5</sub> information is included in this system. The PM<sub>2.5</sub> data is organized in a manner similar to that used to report and retain records from other priority pollutant monitoring. Table 10 includes some of the documents and records that are filed according to the statute of limitations discussed in Section 9.6. In order to access the information as a cohesive unit, all the PM<sub>2.5</sub> information is filed under the major code "PM25", followed by the file names and extensions that lend themselves readily to expansion/addition as the program reaches complete development. The IDEM has two Indianapolis offices as well as three regional offices and one stand-alone support facility located in Porter County Indiana. Each office has a unique responsibility for certain phases of the PM<sub>2.5</sub> monitoring program.

Table 10 PM<sub>2.5</sub> Reporting Package Information

Categories	Record/Document Types	Location
	State Implementation Plan	<sup>1</sup> IGCN
	Reporting agency information	<sup>2</sup> SF, IGCN
	Organizational structure	SF, IGCN
Management and	Personnel qualifications and training	
Management and	Training certification	SF, IGCN
Organization	Quality management plan	SF
	Document control plan	SF
	EPA Directives	SF
	Grant allocations	SF, IGCN
	Network description	SF
Site Information	Site characterization file	SF
Site information	Site maps	SF
	Site pictures	SF
	QA project plans	SF
Eurius u us sutel	Standard operating procedures (SOPs)	SF, <sup>3</sup> NWO, <sup>4</sup> PC
Environmental	Field and laboratory notebooks	SF, NWO, PC
Data Operations	Sample handling/custody records	SF, PC
	Inspection/Maintenance records	SF,PC
Daw Data	Any original data (routine and QC data)	SF
Raw Data	including data entry forms	
	Air quality index report	SF, IGCN
Data Panartina	Annual SLAMS air quality information	SF, IGCN
Data Reporting	Data/summary reports	SF, IGCN, NWO
	Journal articles/papers/presentations	SF, IGCN, NWO
	Data algorithms	SF
Data Managament	Data management plans/flowcharts	SF
Data Management	PM <sub>2.5</sub> data	SF
	Data management systems	SF
	Network reviews	SF
	Control charts	SF, PC
	Data quality assessments	SF
Quality Assurance	QA reports	SF, NWO
	System audits	SF
	Response/Corrective action reports	SF
	Site audits	SF, NOW

<sup>&</sup>lt;sup>1</sup> IGCN - IDEM Office located in the Indiana Government Center North

<sup>2</sup> SF - IDEM Shadeland Office

<sup>3</sup> NWO - IDEM Northwest Regional Office

<sup>4</sup> PC - IDEM Ogden Dunes CAAP Trailer in Porter Co.

### 9.2 Report Submission

### 9.2.1 Quarterly Data Submission

The IDEM ambient monitoring and quality assurance sections submit data to the AQS quarterly and to PARS quarterly. This data is reviewed and submitted to USEPA Region 5 within 90 days from the end of each quarter.

### 9.2.2 Annual Summary Reports Submitted to EPA

As indicated in 40 CFR Part 58, the Indiana Department of Environmental Management submits to the EPA Administrator, through the Region 5 Office, an annual summary report of all the ambient air quality monitoring data from all monitoring stations designated as SLAMS. The report is submitted by July 1<sup>st</sup> of each year for the data collected from January 1<sup>st</sup> to December 31<sup>st</sup> of the previous year. The report contains the following information:

PM-fine  $(PM_{2.5})$ 

Site and Monitoring Information:

- City name (when applicable)
- County name and street address of site location
- AQS- site code
- AQS- monitoring method code

### Summary Data:

- Annual arithmetic mean ( $\mu g/m^3$ ) as specified in 40 CFR Part 50, Appendix N (Annual arithmetic mean NAAQS is  $15\mu g/m^3$ )
- All daily PM-fine values above the level of the 24-hour PM-fine NAAQS (65  $\mu$ g/m<sup>3</sup>) and the dates of occurrence
- Sampling schedule used as once every 6 days, every day, etc

The Office of Air Quality/Ambient Monitoring Branch Chief certifies that the annual summary is accurate. This certification is based on the various assessments and reports performed by the organization, in particular, the Annual QA Report discussed in Section 21 that documents the quality of the PM<sub>2.5</sub> data and the effectiveness of the quality system.

### 9.3 Data Reporting Package Format and Documentation Control

Table 10 represents the documents and records that are filed into the reporting package. All raw data is either collected electronically or on data forms for: the calculation of a  $PM_{2.5}$  concentrations, submission to the AQS database, and QA/QC activities. These data forms are included in the field and analytical methods sections of this chapter. All hard copy information is filled out in indelible ink. Corrections are made by inserting one line through the incorrect entry,

initialing this correction, and placing the correct entry alongside the incorrect entry, if this can be accomplished legibly, or by providing the information on a new line.

#### 9.3.1 Logbooks

Field Logbooks - Logbooks are placed at each sampling site. All field logbooks are bound with numbered pages and are located in the sampler or in close proximity to the sampler. In addition, appropriate data forms for routine operations, inspection and maintenance forms, SOPs and a copy of the R&P Partisol-Plus Air Sampler instruction manual may be taken into the field. Lab Logbooks - Logbooks have also been issued for use in the laboratory. Lab logbooks are bound with numbered pages. One logbook has been designated for general comments/notes; others are associated with, the temperature and humidity recording instruments, the refrigerator, calibration equipment/standards, and the analytical balances used for this program. In addition to bound logbooks, various computer logs are maintained (i.e., spreadsheet files). Appropriate steps are taken to ensure the integrity of all computer files.

# 9.4 PM<sub>2.5</sub> Records to Create and Retain

Additional requirements for the preparation and retention of documentation are found in the related SOPs included as appendixes of this document, CFR Part 50, and 53, and the R&P Partisol-Plus Air Sampler operator's instruction manual. The sections to follow summarize these requirements.

#### 9.4.1 Sampler Siting and Maintenance Records

Documentation regarding siting and maintenance of the  $PM_{2.5}$  samplers are retained in the files. These include the following:

- Site selection criteria checklist; multidirectional photographs of each site with the PM<sub>2.5</sub> sampler in the foreground, a topographical map of the area, and a copy of the original site documentation records. For new PM<sub>2.5</sub> monitoring sites, these site documentation records will also be submitted for the AQS database.
- Procurement log for field equipment, (see example below) and acceptance/rejection test results (see example below):

# **Example Procurement Log**

			PO		D	ate			Accept/	
Item	Description	Qty	#	Vendor	Ord.	Rec'd.	Cost	Initials	Reject	Comments
1 case filters	2 µm pore 46.2 mm diameter	60	971- 100	WIZ Supply	8/1/98	8/15/98	\$100	ABC	Accept	Examined 8/20/98

Table 11
Acceptance Checks and Limits for Procurement of Equipment and Supplies

Equipment	Acceptance check	Acceptance limits	Action if requirements are not met
Field operations			
Sampler	Sampler and accessories complete; no evidence of damage. Model designated as reference or equivalent method. Pump and display work.	Specifications outlined in 40 CFR Part 50, Appendix L	Reject sampler
Calibration quality assurance/quality control (QA/QC) equipment for flow rate, temperature, pressure, etc.	Accompanied by certificate. Check values against (NIST)-traceable standards.	Within accuracy limits in this document	Adjust or reject Equipment
Audit equipment	Same as for calibration equipment, but must not be the same equipment.	Within accuracy limits in this document	Adjust or reject Equipment

- Warranty and maintenance records for each PM<sub>2.5</sub> sampler. A maintenance logbook is kept for each sampler and continually updated.
- Manufacturer-supplied calibration and traceability records for thermometers flow rate
  measuring devices, and pressure sensors used for calibrating, checking or auditing PM<sub>2.5</sub>
  samplers are maintained and filed in the Quality Assurance Laboratory at the Shadeland
  facility and according to SOPs.

# 9.4.2 Analytical Laboratory Installation Records

Records have been established regarding the physical set-up of the analytical laboratory and include at least the following:

- Equipment inventory (microbalance, antistatic devices, calibration and check weight etc.)
- Procurement log and notes on acceptance/rejection tests (see the example below):

Table 12
Acceptance Checks and Limits for Procurement of Equipment and Supplies

Equipment	Acceptance check	Acceptance limits	Action if requirements not met
Filters, Teflon	Of correct type and undamaged.	Type as described in (40 CFR Part 50, App L)	Reject filters
Filter cassettes	Of correct type & undamaged.	As specified by sampler manufacturer	Reject filter Cassettes
Filter/cassette protective containers	Of correct type and undamaged.	As described in this document	Reject protective Containers
Filter-handling containers	Of correct type & undamaged.	As described in this document	Reject filter-handling Containers
Analytical microbalance	Accompanied by certificate; check values against working standards.	Readability 1 $\mu$ g, Repeatability 1 $\mu$ g	Adjust or reject Equipment

• Daily records are kept to demonstrate that the temperature and relative humidity within the clean room are held within specified limits.

#### 9.4.3 Field Sampling Operation Records

PM<sub>2.5</sub> sampling requires that numerous paper and computerized records be reviewed, reported and filed. These records should include at least the following:

• Sampler calibration logbooks or data sheets for entering results of temperature, pressure, and flow rate checks, audits, and calibrations. Records are also maintained on all associated equipment to insure proper traceability and certification. A separate logbook or file is maintained for each sampler and related piece of equipment used in the network. These logbooks/files include the results of the sampling procedure checks like those listed in Table 13 below:

Table 13 Sampling Procedure Checks

Procedure	Frequency & Method	Requirements	Action if requirements not met
Filter installation	Check filter. Install filter in cassette, close filter holder.	Filters must be ID'ed, tare- weighed, undamaged, & in cassette.	Void the filter, and install substitute filter/cassette.
Sample validation & documentation	Check each sample and the keypad display or downloaded sampler data for completeness.	Record sampling date, filter and sampler ID, station location, flow rates, sample time, & unusual conditions on data sheet or computer screen.	Complete or correct documentation. Cross out invalid information with a single line. Initial & date changes.
Post sample inspection	Check filters in cassettes for tears, missing pieces, or leakage. Review sampler operation.	There should be no evidence of filter damage or sampler malfunction.	Flag sample as questionable; correct cause of malfunction.
Leak checks	Check for leaks. When suspected, or after every fifth sample day service of WINS.	Leak-check results must be within parameters specified by manufacturer.	Determine cause of leak & correct. Validate and/or calibrate sampler flow rate.
Flow rate checks	Check flow rate at least every 4 weeks or once per month at each collector in the network.	Indicated sampler flow rate must be within ±4 percent of the measured flow rate.	Correct problems. Recalibrate sampler if needed.
Field blank check	At least one field blank available per weighing session. Install, then immediately remove filter from sampler; store in protective container inside sampler case. Or install in idle single filter sampler for 24 hours, then remove & process. For sequential samplers, install in unused holder.	Rotate from sampler to sampler so all are checked. Special requirements for sequential samplers. Should have one or more field blanks with each batch of filters to be weighed. Refer to Section 7.0.	Reassess filter handling techniques and storage conditions.

- A sample run data sheet is completed by the site operator who will keep a copy for the site field site record and the original run data sheet will accompany the filter sample to the laboratory.
- A PM<sub>2.5</sub> Flow Check sheet displayed in Form 1 is maintained by the site operator.

# Form 1 Single Filter PM<sub>2.5</sub> Data Sheet

Site Identification/Name:							
LABORATORY INFORM							
Laboratory Operator initials: Sample Frequency (circle): 1/1 1/3							
Filter ID:	Cassette ID:	Visi	t Date:				
Filter Information:	Initial Weight:	mg Date: _					
	Final Weight:	mg Date: _					
$Conc. = \frac{Wt. Gain}{Volume} * 1000$	Calculated Concentr	ation:	_µg/m <sup>3</sup>				
Sample Receipt Date	te/Time:						
Min/Max Temp. of	Filter During Transport:	/	°C Initials:				
Sample Start Date:	Sam	ple Stop Date:					
FIELD INFORMATION:							
Sample Set-up:							
Date/Time Sample Set-up:_	<u></u>						
Date/Time Sample Set-up:/ Set-up Operator:							
WINS #: <u>200FA</u>							
Conditions at Time of Samp		$T_o = ambient temp.$					
_	·	_	D·				
	$\begin{array}{cccccccccccccccccccccccccccccccccccc$						
(Ambient pressure must be	within ±10 mmHg and an	nbient temperature must be	within ±2 °C)				
Sample Pickup:							
Date/Time Sample Pickup:/ Pick-Up Operator:							
Final Conditions of Sample	Pick-up (as retrieved from	m 5 data storage screens):					
Valid Elapsed Time:	<u>hr</u> Volume	m <sup>3</sup> % CV					
	Minimum	Average	Maximum				
Ambient Temp. (Amb T)	°C	°C	°C				
Ambient Pressure (Pres)	mmHg	mmHg	mmHg				

Any status conditions or power failure should be recorded in notes as well as in the monitor logbook

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Was Sample Shipped Cold? YES or NO (If same	ple is questionable, please	explain in notes)
Monitor in WAIT or SAMPLING mode prior to leaving site?	YES	NO
Air supply hose reconnected to the Supply magazine?	YES	NO
WINS Impactor and First Stage Inlet installed and secure?	YES	NO
Temperature probes installed and secure?	YES	NO
All procedures recorded in monitor logbook?	YES	NO
NOTES:		

# 9.4.4 Laboratory Operations Records

Data records from activities in the weighing/clean room include, at a minimum, the following:

- Records of lab temperature and humidity control
- Lab data forms are completed and retained. These forms are found in the Analytical SOP.
- Internal Quality Control logbook
- Results of calibrations and servicing of the Mettler UMT2
- Results of filter integrity checks and determinations of the conditioning periods required for various filter batches
- Completed PM<sub>2.5</sub> sampler run data sheet
- Records of sample numbers and location of archived PM<sub>2.5</sub> samples

# 9.4.5 Quality Assurance Records

Documentation of quality assurance systems and performance audits are maintained and the following information:

- Flow rate, temperature, and pressure audit data entered and stored in the Office of Air Management Database (OAMD). In addition, paper audit sheets are used initially to record audit data. An example audit sheet is included in the QA Audit SOP.
- Systems audit questionnaire and subsequent applicable audit sheets (as found in the QA Audit SOP).

Accuracy and precision audit result records are filed in Indianapolis Shadeland Laboratory Shadeland Office as well as being submitted to the EPA Regional Office and to the AQS data system.

#### 9.5 Electronic Data Collection

All R&P Partisol-Plus Air Samplers used in Indiana's PM<sub>2.5</sub> Ambient Air Quality Monitoring Program are equipped with software and a 9 to 9 pin computer cable to enable the transfer of data files containing stored information to a personal computer. Samplers at some sites are connected to a phone line and perform data downloads directly to the database at the Shadeland facility. Downloading/uploading information is found in the appropriate Ambient Air Monitoring Section SOP and the R&P Partisol-Plus Air Sampler instruction manual. In order to reduce the potential for data entry error, field operators continue to provide a hard copy back-up of automated data, even after the software is operational. This hard copy backup continues until a system review determines that data entry error is insignificant.

In March of 2000, an automated system was instituted that documents all aspects of filter information and data. The Analytical Section uses the Gravimetric Laboratory Information System (GLIMS $^{\text{TM}}$ ). GLIMS is an Access database which interfaces to the lab's microbalance

which allows, for example, automated data collection of initial and final filter weights, sampler run data, quality control data and quality assurance data. Through the programs main toolbar, the lab technician produces and prints bar codes for filter containers, records tare weights of unexposed filters, re-weighs and records exposed filters, calculates net weights, and finally, produces a number of configurable reports.

#### 9.6 Data Reporting Package Archiving and Retrieval

As stated in 40 CFR Part 50, Appendix L. CFR part 31.42, in general, all the information listed in Table 10 and discussed throughout Section 9, is retained for 3 years. However, if any litigation, claim, negotiation, audit or other action involving the records has been started before the expiration of the 3-year period, the records are retained until completion of the action and resolution of all issues which arise from it, or until the end of the regular 3-year period, whichever is later. The IDEM will extend this regulation in order to store records for three full years past the year of collection. For example, any data collected in calendar year 1999 (1/1/99 - 12/31/99) are retained until, at a minimum, January 1, 2003; unless the information is used for litigation purposes.

# 10.0 Sampling Design

This section describes all of the relevant components of the SLAMS gravimetric mass  $PM_{2.5}$  monitoring network being operated by the IDEM, including the network design for evaluating the quality of the data. This entails describing the key parameters to be estimated, the rationale for the locations of the  $PM_{2.5}$  monitors and the QA samplers, the frequency of sampling at the primary and QA samplers. The network design components comply with the regulations stipulated in 40 CFR Part 50, Appendix L, CFR Part 58 Section 58.13, Appendix A, and Appendix D.

#### 10.1 Scheduled Project Activities, Including Management Activities

IDEM monitors PM<sub>2.5</sub> concentrations at 32 locations as of January 2003 with 4 sites equipped for collocated sampling (duplicate samplers). The Indianapolis Office of Environmental Services also monitors PM<sub>2.5</sub> concentrations at 7 locations in Marion County with 1 site equipped for collocated sampling.

# 10.2 Rationale for the Design

#### **10.2.1 Primary Samplers**

The primary purpose of Indiana's  $PM_{2.5}$  Ambient Air Quality Monitoring Program is to measure compliance with national standards for particulates less than or equal to 2.5 micrometers. These standards are based on 24-hour average  $PM_{2.5}$  concentrations, and summarized as:

- 1. The three-year average of the annual 98th percentiles of  $PM_{2.5}$  concentrations at any population-oriented monitoring site is not to exceed 65  $\mu$ g/m<sup>3</sup>.
- 2. The three-year average of the annual mean of PM<sub>2.5</sub> concentrations is not to exceed 15 μg/m<sup>3</sup>. The average may be based on a single community-oriented monitoring site or may be based on the spatial average of community-oriented monitoring sites in a community monitoring zone (CMZ).

The key characteristics being measured are annual 98th percentiles and annual means of twenty-four hour average PM<sub>2.5</sub> concentrations.

By employing R&P Partisol-Plus Air Sampler, Indiana is assured to be measuring the PM<sub>2.5</sub> concentrations with regard to evaluating compliance with the PM<sub>2.5</sub> NAAQS. By complying with the sampling frequency requirements of (40 CFR Part 50, Appendix L) CFR Part 58 Section 58.13, Indiana assumes that the sampling frequency is sufficient to attain the desired confidence in the annual 98th percentile and annual mean of PM<sub>2.5</sub> concentrations in the vicinity of each monitor. By selecting sampler locations using the rules in (40 CFR Part 50, Appendix L) CFR Part 58 Appendix D, the IDEM is confident that the PM<sub>2.5</sub> concentrations within its jurisdiction are adequately characterized.

# 10.2.2 QA Samplers

The purpose of collocated (duplicate) samplers and the FRM performance evaluation is to estimate the precision and bias of the various PM <sub>2.5</sub> samplers. The DQOs section of this document state that, for a 3-year period, the concentrations measured by a sampler must be within ±10% of the true concentration as measured by a FRM sampler and that the coefficient of variation of the relative differences must be less than 10%. These levels of bias and precision need to be accomplished so that decision makers can make decisions about attainment and/or non-attainment of the PM <sub>2.5</sub> NAAQS with sufficient confidence. To estimate the level of bias and precision being achieved in the field, some of the sites will operate collocated samplers and some of the sites are audited using FRM samplers.

If a sampler is operating within the required bias and precision levels, then the decision maker can proceed knowing that the decisions are supported by unambiguous data. If, however, a sampler exceeds either the bias limits or the precision limits or both, then the decision maker cannot use the data to make decisions at the desired level of confidence and corrective action must be implemented to ensure that future data collected by the sampler does meet the bias and precision limits. Thus the key characteristics being measured with the QA samplers are bias and precision.

To determine whether these characteristics are measured with sufficient confidence, IDEM addresses sampler type, sampling frequency, and sampler siting for the QA network. As with the primary PM<sub>2.5</sub> network, by using R&P Partisol-Plus Air Samplers, maintaining the sampling

frequency specified in 40 CFR Part 50, Appendix L, CFR Part 58 Appendix A, and collocating the number of samplers as specified in 40 CFR Part 50, Appendix L, CFR Part 58 Appendix A, the IDEM assumes its QA network will measure bias and precision with sufficient confidence. These issues are described in more detail in Section 10.4.

#### 10.3 Design Assumptions

Sampling design is based on the assumption that following the rules and guidance provided in the CFR will result in data that can be used to measure compliance with the national standards. The only issue at IDEM's discretion is the sampler siting, and to a degree, sampling frequency. The siting assumes homogeneity of PM<sub>2.5</sub> concentrations within community monitoring zones (CMZ) and heterogeneity between CMZ. Monitoring planning area (MPA) and CMZ boundaries are regularly reviewed, as part of the network reviews. The basis for creating and revising the boundaries is described in the following section.

# 10.4 Procedure for Locating and Selecting Environmental Samples

#### **10.4.1 Primary Samplers**

The SLAMS PM<sub>2.5</sub> network design meets one of six basic monitoring objectives, described in 40 CFR Part 50, Appendix L, CFR Part 58, Appendix D. These are:

- 1. Determine the highest concentrations expected in the area covered by the network.
- 2. Determine representative concentrations in areas of high population density.
- 3. Determine the impact on ambient pollution levels of significant sources or source categories.
- 4. Determine general background concentrations levels.
- 5. Determine the extent of regional pollution transport among populated areas.
- 6. In support of secondary standards, determine the welfare-related impacts in more rural and remote areas.

The procedure for siting the PM<sub>2.5</sub> samplers to achieve the six basic objectives has been based on judgmental sampling, as is the case for most ambient air monitoring networks. Judgmental sampling uses data from existing monitoring networks, knowledge of source emissions and population distribution, and inference from analyses of meteorology to select optimal sampler locations.

The Indiana Department of Environmental Management, Air Monitoring Branch operates the  $PM_{2.5}$  monitoring network throughout the state with exception of the city of Indianapolis Office of Environmental Services (IOES). The IOES operates the  $PM_{2.5}$  monitoring network and data reporting for Marion County and the greater Indianapolis metro area. The number of SLAMS sites and their location was determined based upon the information provided in 40 CFR Part 50, Appendix L, CFR Part 58 Appendix D and in *Guidance for Network Design and Optimum Site Exposure for PM*<sub>2.5</sub> and  $PM_{10}$ .

# 11.0 Sampling Methods and Requirements

Indiana uses Rupprecht & Patashnick Partisol-Plus Model 2025 Sequential Air Samplers to meet the measurement goals of the PM <sub>2.5</sub> Ambient Air Quality Monitoring Program. Monitoring stations sample either a one in three day or a one in six day schedule. Daily (one in one day) sampling was discontinued at the end of 2002.

All filters used in state agency operated PM <sub>2.5</sub> Ambient Air Quality Monitoring Program are conditioned, weighed, and loaded into filter cassette assemblies (storage magazines) in the Office of Air Quality's Analytical laboratory in Indianapolis prior to distribution into the field. Exposed filters are returned to the Office of Air Quality's laboratory for post sampling conditioning and analysis. This process is documented to ensure no data is lost due to inadequate or improper handling of the sampling media.

Additional information regarding permissible sample holding times is specified in 40 CFR Part 50, Appendix L, Section 2.12 of the U.S.EPA QA Handbook, the Indiana Department of Environmental Management's SOP on filter transport, and Table 16 of this document.

# 11.1 Support Facilities for Sampling Methods

Most field sites in Indiana's  $PM_{2.5}$  monitoring program have little or no indoor support facility. They are stand alone or roof top locations without a monitoring trailer.

Therefore, operators are equipped with portable sampling kits consisting of:

- fuses
- temperature verification/audit/calibration standard
- flow rate adaptor
- flow rate verification/audit/calibration standard
- flow rate verification filter
- sampler operator/service manual
- PM<sub>2.5</sub> sampling and field maintenance SOPs
- impactor oil
- cleaning wipes
- spare rain collector
- filter data sheets
- miscellaneous tools
- voltmeter
- palm top computer and download cable
- spare WINS impactor
- sample magazines
- filter transport container
- field blanks

# 11.2 Sample Set-up

All filters are kept in protective cassette cartridges/magazines until installation in the sampler. Filter cassette magazines may be installed or exchanged in any of the sampler's operating modes.

The R&P Partisol-Plus Sampler allows magazines for supply and storage. Any scheduled maintenance (i.e. impactor replacement, pressure and temperature checks etc.) is performed prior to the installation of a new filter storage magazine. The only holding time that affects sample setup is the 30 day window from the time a filter is pre-weighed to the time it is installed in the monitor. At collocated sites the second monitor is set up to run at a sample frequency of 1 in 6 days; sample set-up takes place on the same day as the primary (reporting) sampler. Detailed sample set-up procedures are available in the IDEM PM<sub>2.5</sub> Calibration/Verification/Sample Set-up for Rupprecht & Patashnick Partisol-Plus Model 2025 Sequential Air Samplers standard operating procedure. In general the procedure is:

- 1. Annotate the following on a data sheet:
  - Date and time of the sampler set-up visit
  - Site designation and location
  - Sampler model, ID number, and filter ID number
  - Sample start date and time
  - Current ambient temperature and barometric pressure indicated by the sampler
  - Unusual conditions that may affect the samples (e.g. construction activity, weather)
  - Set-up operator's signature or initials

Note: Each data sheet is individually labeled with a bar code sticker printed with a site name, site code number and sample run month/day/year.

- 2. Open the sampler enclosure.
- 3. Ensure proper placement of the filter magazines. Supply magazine is on the left side and the storage magazine is on the right side.
- 4. Ensure that the air connection fitting of the cassette magazine is facing toward the operator and attach the magazine using the mounting studs on the sampler.
- 5. Lock the magazine into place.
- 6. Remove and cap the filter storage magazine (right) and replace it with an empty storage magazine.
- 7. Prepare the spent filter magazine for transport back to the laboratory facility in accordance with the SOP.

#### 11.3 Post Sampling Operations/Sample Recovery

Recovery of each filter from a sampler in the IDEM network must be performed within 168 hours (7 days) from the end of the sample period for that filter. For one in three (1/3) day sampling, sample recovery is normally on the day after the second sample is collected. The next sample set-up for two samples also takes place on this day. At collocated sites, the sample from the second monitor is recovered on the same day as the primary sampler. Since the IDEM PM<sub>2.5</sub> monitoring network uses sequential samplers, sites with the 1/3 day sampling frequency will most often require only one site visit a week, except for one week out of every six weeks, where two site visits are required. Sample recovery procedures are available in the IDEM PM<sub>2.5</sub> Calibration/Verification/Sample Set-up for Rupprecht & Patashnick Partisol-Plus Model 2025 Sequential Air Samplers standard operating procedure. In general the procedure is:

- 1. Inspect the sampler and its screen display to ensure proper operation. If problems are apparent, explain on the sample run sheet and perform corrective action according to the R&P Partisol-Plus Air Sampler instruction manual or the appropriate Ambient Monitoring Section SOP.
- 2. Record the following on the sampler data sheet. This information is also downloaded via a laptop or palmtop PC to an electronic file.
  - end operator's initials
  - date and time of sample pick-up
  - stop time and total elapsed time
  - final flow rate, average flow rate, coefficient of variation of the flow rate, and total volume sampled
  - indicated barometric pressure and temperature at the end of the run
  - note any flags triggered by the sampler (e.g. power outage, flow rate variation)
  - explanation of any voided or questionable data
  - note any unusual field occurrences (e.g. road construction)
  - download data according to the R&P Partisol-Plus Air Sampler operations manual
  - remove and cap the filter storage magazine and prepare for transport according to the ambient monitoring SOP to be included as an appendix to this document
  - perform any scheduled maintenance activities
  - install a supply magazine if necessary
  - note unusual conditions that may affect particle loading of the sample(s)
  - secure the site (lock doors, gates, store ladder, etc.) before departure

# 11.4 Sampling/Measurement System Corrective Action

Corrective action measures in the  $PM_{2.5}$  Air Quality Monitoring Network are taken to ensure the data quality objectives are attained. There is the potential for many types of sampling and measurement system corrective actions. Table 14 lists some of the expected problems and corrective actions needed for the IDEM to maintain a well-run  $PM_{2.5}$  network.

# Table 14 Field Corrective Action

Item	Problem	Action	Notification
Filter Inspection (Pre-sample)	Pinhole(s) or torn	<ol> <li>If additional filters have been brought, use one.</li> <li>Void filter with pinhole or tear.</li> <li>Use new field blank filter as sample filter.</li> <li>Obtain a new filter from lab.</li> </ol>	Document on field data sheet.     Document on field data sheet.     Notify Field Manager.
Filter Inspection (Post-sample)	Torn or otherwise suspect particulate by pass 46.2 mm filter.	<ol> <li>Inspect area downstream of filter holder &amp; determine if particulate has bypassed filter.</li> <li>Inspect in-line filter before sample pump &amp; determine if excessive loading has occurred. Replace as necessary.</li> </ol>	Document on field data sheet.     Document in logbook.
Sample Flow Rate Verification	Out of Specification (±4% of transfer standard)	<ol> <li>Remove flow rate device, re-connect, and perform flow rate check.</li> <li>Perform leak test.</li> <li>Check flow rate at 3 points (15.0 lpm, 16.7 lpm, and 18.3 lpm) to determine if flow rate problem is with zero bias or slope.</li> <li>Re-calibrate flow rate.</li> </ol>	<ol> <li>Document on data sheet.</li> <li>Document on data sheet.</li> <li>Document on data sheet.</li> <li>Notify Field Manager,</li> <li>Document on data sheet.</li> <li>Notify Field Manager.</li> </ol>
Leak Test	Leak outside acceptable tolerance (80 ml/min)	Remove flow rate device and perform leak test.     Inspect all seals and O-rings, replace as necessary and perform leak test.     Check sampler with different leak test device.	<ol> <li>Document in logbook.</li> <li>Document in logbook, notify</li> <li>Field Manager, &amp; flag data since last successful leak test.</li> <li>Document in logbook &amp; notify</li> <li>Field Manager.</li> </ol>
Sample Flow Rate	Consistently low flows documented during sample run	<ol> <li>Check programming of sampler flow rate.</li> <li>Check flow with a flow rate verification filter &amp; determine if actual flow is low.</li> <li>Inspect in-line filter downstream of filter, replace as necessary.</li> </ol>	Document in logbook.     Document in logbook.     Document in logbook.
Ambient Temperature Verification, and Filter Temperature Verification.	Out of Specification (±4 °C of standard)	Ensure thermocouples are immersed in same liquid at same point without touching sides or bottom of container.     Use ice bath or warm water bath to check a different temperature. If acceptable, re-perform ambient temperature verification.     Connect new thermocouple.     Check ambient temperature with another NIST traceable thermometer.	Document on data sheet.     Document on data sheet.     Document on data sheet. Notify Field Manager.     Document on data sheet. Notify Field Manager.
Elapsed Sample Time	Out of Specification (1 min/mo)	Check Programming, Verify Power Outages	Notify Field Manager.
Elapsed Sample Time	Sample did not run	Check Programming     Program sample run to start while operator is at site. Use a flow verification filter.	Document on data sheet.     Notify Field Manager.     Document in logbook. Notify Field Manager.
Power	Power Interruptions	Check Line Voltage.	Notify Field Manager.
Power	LCD panel on, but sample not working	Check circuit breaker, some samplers have battery back-up for data but will not work without AC power.	Document in logbook.
Data Download	Data won't transfer to laptop computer	Document key information on sample data sheet.  Make certain problem is resolved before data is written over in sampler microprocessor.	Notify Field Manager.

# 11.5 Sampling Equipment, Preservation, and Holding Time Requirements

This section details the techniques employed to prevent sample contamination, the volume of air to be sampled, temperature preservation requirements, and the permissible holding times to ensure against degradation of sample integrity.

### 11.5.1 Sample Contamination Prevention

Indiana's PM<sub>2.5</sub> network has filter handling requirements that are designed to minimize sample contamination. Powder free gloves are worn while handling filters. Filters conditioned in the "clean room" are stored in open petri dishes. Once they are weighed, they are returned to a bar coded lidded petri dish. These petri dishes then remain sealed until the lab technician places each filter into a cassette. Each filter cassette is then placed into a sampling magazine.

After exposure, the filter cassettes are placed in storage magazines (automatically by the sampler). The site operator places storage magazines containing the exposed filter cassettes into padded metal containers provided by the manufacturer. Coolers equipped with blue ice and a maximum/minimum thermometer are used for transport of the storage magazines back to the Indiana Department of Environmental Management, Office of Air Quality laboratory. Once samples are weighed, they are stored with the particulate side up in sealed and labeled petri dishes.

#### 11.5.2 Sample Flow Rate and Volume

The flow rate is specified in 40 CFR Part 50, Appendix L at 16.67 liters per minute (l/min). Since capture of the fine particulate is predicated upon a design flow rate of 16.67 l/min, deviations of greater than 10% from the design flow rate enable a shut-off mechanism for the sampler. Based on the design flow rate the total volume of air collected over a 24-hour sampling interval is 24 cubic meters (m³). Samples are normally collected over a 24-hour period; however, in some cases a shorter sample period may occur due to events such as monitor maintenance or audit during a sampling period. If a sample period is less than 23 hours or greater than 25 hours, the sample is flagged and the QA Officer notified. All sample concentrations are calculated using the elapsed time recorded by the sampler.

# 11.5.3 Temperature Preservation Requirements

The temperature requirements of the  $PM_{2.5}$  network are specified in 40 CFR Part 50, Appendix L. During transport from the weigh room to the sample location, there are no specific requirements for temperature control; however, the filters are contained is sealed petri dishes and protected from excessive heat/cold.

Table 15
Filter Temperature Requirements

Item	Temperature Requirement	Reference
Filter temperature control during	No more than 5 °C above ambient	(40 CFR Part 50, Appendix L)
sampling and until recovery.	temperature.	Section 7.4.10
Filter temperature control from time	Protected from exposure to	(40 CFR Part 50, Appendix L)
of recovery to start of conditioning.	temperatures over 25 °C.	Section 10.13
Post sample transport so that final		(40 CFR Part 50, Appendix L)
weight may be determined up to 30	4 °C or less	Section 8.3.6
days after end of sample period.		Section 6.3.0

# 11.5.4 Permissible Holding Times

IDEM adheres to the permissible holding times for the PM<sub>2.5</sub> samples which are clearly detailed in both 40 CFR Part 50, Appendix L, *and Section 2.12 of the U.S. EPA QA Handbook*. Holding times are listed in Table 16.

Table 16 Holding Times

Item	Holding Time	From:	To:	Reference
Pre-weighed Filter	<30 days	Date of Pre-weigh	Date of Sample	40 CFR Part 50, Appendix L, Section 8.3.5
Recovery of Filter	≤196 hours	Completion of sample period	Time of sample recovery	40 CFR Part 50, Appendix L, Section 10.10
Transport of Filter	<24 Hours (ideally)	Time of recovery	Time placed in conditioning room	40 CFR Part 50, Appendix L, Section 10.13
Post Sample Filter stored at <4 °C.	<u>&lt;</u> 30 days	Sample end date/time	Date of Post Weigh	40 CFR Part 50, Appendix L, Section 8.3.6
Post Sample Filter continuously stored at <25 °C.	≤10 days	Sample end date/time	Date of Post Weigh	40 CFR Part 50, Appendix L, Section 8.3.6

#### 12.0 Chain-of-Custody

It is important to keep good records in any air pollution monitoring program. This is even more significant when dealing with  $PM_{2.5}$  because the reference or Class I equivalent samplers are manual methods that require transfer and handling of the data and samples by several people. IDEM maintains records for all stages of the sampling process that includes: pre-sampling, post sampling, filter receipt, and filter archiving.

# 12.1 Pre-Sampling Chain-of-Custody

Logbooks and computerized records (GLIMS<sup>TM</sup>/Access database) are maintained at the Indiana

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Department of Environmental Management, Office of Air Quality, Air Monitoring Branch. This documentation includes; significant dates, responsible party and intended destination (i.e. storage, local agency, regional office, etc.) as well as clean room conditions for the following phases:

- initial receipt to the laboratory
- pre weighing conditioning
- initial tare weight
- quality assurance re-weighs (establishes accuracy of the measurement)
- field distribution

# 12.2 Field Chain-of-Custody

Site operators are responsible for determining the condition and intended sampling location of filters received for field use from the Indiana Department of Environmental Management, Office of Air Quality. This includes verifying that seals on shipping and storage containers are intact and that filter IDs on data sheets correspond with filter IDs on each sampling unit.

# **12.3 Post Sampling Chain-of-Custody**

Logbooks and computerized records (GLIMS<sup>TM</sup>/Access database) are maintained in the IDEM, OAQ laboratory for the documentation of filters. The logbooks include the following:

- post sampling receipt to the laboratory facility
- post sampling conditioning
- post sampling initial weighing
- subsequent post sampling re-weighs (establishes stability)
- post sampling quality assurance re-weighs (establishes accuracy of the measurement)

#### 12.4 Archiving Chain-of-Custody

After completion of the post sampling weighing activities, filters are stored for at least one year past the date of sampling. SOP's for this storage are included as appendixes to this chapter.

#### 13.0 Analytical Method Requirements

Concentrations of  $PM_{2.5}$  in Indiana's ambient air are determined by the gravimetric analysis of filters obtained from the operation of R&P Partisol-Plus Air Samplers. The net weight gain of a sample is calculated by subtracting the initial (tare) weight of the filter from final weight (post-sampling weight). This weight gain, divided by the total sample airflow, is used to calculate the concentration of ambient  $PM_{2.5}$  reported as  $\mu g/m^3$ . Since the method is non-destructive, and due to the possible interest in sample composition, the filters are archived after the gravimetric analysis.

In order to provide high quality data, laboratory conditions are closely monitored. Staff maintain strict attention to detail, both in their weighing technique and the microbalance operation and maintenance.

#### 13.1 Sample Preparation

In January of 2003, the approximate number of sample filters that are prepared, conditioned, and weighed is approximately 140 per week. In addition, field blanks, lab blanks, and flow check filters must also be prepared. Indiana's PM<sub>2.5</sub> monitoring frequency is summarized below:

Sample Frequency	No. of Samplers
1/3	32
1/6	4

Upon delivery of approved 46.2 mm Teflon filters for use in the IDEM network, the receipt is documented and the filters are stored in the conditioning/weighing room/laboratory "clean room". Storing filters in the laboratory makes it easier to maximize the amount of time available for conditioning. Upon receipt, cases of filters are labeled with the date of receipt, opened one at a time, and used completely before opening another case. All filters in a lot are used before a case containing another lot is opened. When more than one case is available to open, the "First In - First Out" rule applies.

Filters are taken out of the case when there is enough room for in the pre-sampling weighing section of the clean room. Filters are inspected according to the FRM criteria to determine compliance. Filters are then stored in the clean room in labeled petri dishes. The minimum conditioning period is 24 hours. Filters are not left out for excessive periods of conditioning since some settling of dust is possible.

#### 13.2 Analysis Method

#### 13.2.1 Analytical Equipment and Method

IDEM uses a Mettler Model UMT2 microbalance for the gravimetric analysis of the  $PM_{2.5}$  samples. This instrument meets all criteria set forth in CFR Part 50, Appendix L and is maintained under agreement with the manufacturer. Two sets of Troemner weights are used as primary and working standards. The detailed procedure for the handling and analysis of  $PM_{2.5}$  samples is found in the laboratory SOP.

Both pre and post sample filters are weighed on the same Mettler UMT2 balance and all weighing is done by the same laboratory personnel whenever possible. Refer to the table below for additional filter preparation and analysis checks.



**Photo 2 Filter Weighing** 

# Table 17 Filter Preparation and Analysis Checks

Activity	Method and Frequency	Requirements	Action if the requirements are not met
Microbalance Use		Resolution of 1 $\mu$ g, repeatability of 1 $\mu$ g.	Obtain proper microbalance.
Control of bal. environment		Climate-controlled, draft-free room or chamber or equivalent.	Modify the environment.
Use of Mass reference standards	Working standards checked every 3 to 6 months against laboratory primary standards.	Standards bracket weight of filter, individual standard's tolerance less than 25 $\mu$ g, handle with smooth, nonmetallic forceps.	Obtain proper standards or forceps.
Filter handling	Observe handling procedure.	Use powder-free gloves and smooth forceps. Replace <sup>210</sup> Po antistatic strips every 6 months.	Discard mishandled filter or old antistatic strip.
Filter integrity check	Visually inspect each filter.	No pinholes, separation, chaff, loose material, discoloration, or filter nonuniformity.	Discard defective filter.
Filter identification	Write filter number on filter handling container, sampler number on protective container, and both numbers on laboratory data form in permanent ink.	Make sure numbers are written legibly.	Replace label or correct form.
Pre-sampling filter equilibration	Determine the correct equilibration conditions and period (at least 24 hours) for each new lot of filters. Observe and record the equilibration chamber relative humidity and temperature; enter to lab data form.	Check for stability of laboratory blank filter weights. Weight changes must be $<15 \mu\mathrm{g}$ before and after equilibration. Mean relative humidity between 30 and (40 CFR Part 50, Appendix L) percent, with a variability of not more than $\pm 5$ percent over 24 hours. Mean temperature is held between 20 and 23 °C, with a variability of not more than $\pm 2$ °C over 24 hours.	Revise equilibration conditions and period. Repeat equilibration.
Pre-sampling filter equilibration	Determine the correct equilibration conditions and period (at least 24 hours) for each new lot of filters. Observe and record the equilibration chamber relative humidity and temperature; enter to lab data form.	Check for stability of laboratory blank filter weights. Weight changes must be $<15~\mu g$ before and after equilibration. Mean relative humidity between 30 and (40 CFR Part 50, Appendix L) percent, with a variability of not more than $\pm 5$ percent over 24 hours. Mean temperature is held between 20 and 23 °C, with a variability of not more than $\pm 2$ °C over 24 hours.	Revise equilibration conditions and period. Repeat equilibration.
Initial filter weighing	Observe all weighing procedures. Perform all QC checks.	Neutralize electrostatic charge on filters. Wait long enough so that the balance indicates a stable reading (oscillates no more than $\pm 2$ , drifts no more than 3 $\mu$ g, in 5-10 sec).	Repeat weighing.
Internal QC	After approximately every tenth filter, zero the microbalance and reweigh the two working standards. Weigh three laboratory filter blanks. Reweigh one duplicate filter with each sample batch (duplicate weighing).	The working standard measurements must agree to within $3 \mu g$ of the certified values. The blank and duplicate measurements must agree to within $15 \mu g$ .	Flag values for validation activities.
Post-sampling inspection, documentation and verification	Examine the filter and field data sheet for correct and complete entries. If sample was shipped in a cooled container, verify that low temperature was maintained.	No damage to filter. Field data sheet complete. Sampler worked OK.	Notify Lab Manager. Discard filter. Void sample.

#### 13.2.2 Conditioning/Weight Room

A room or "clean room" specifically designed for the conditioning and weighing of the PM<sub>2.5</sub> samples has been constructed at the Indiana Department of Environmental Management, Office of Air Quality. This clean room is a restricted access area and it meets the criteria in *CFR Part* 50, Appendix L and Section 2.12 of the Quality Assurance Handbook for Air Pollution Measurements Systems. These criteria include:

- mean temperature of 20-23 °C
- temperature controlled to within ±2 °C over 24 hours
- mean humidity 30-40%
- humidity control ±5 % relative humidity over 24 hours
- temperature and relative humidity is continuously monitored with 1-minute averages, collected by a data logger and information saved to a computer hard drive

#### 13.2.3 Environmental Control Requirements

The temperature requirements of the PM<sub>2.5</sub> network are explicitly detailed in 40 CFR Part 50, Appendix L, CFR Part 50. In the weigh room laboratory, the filters must be conditioned for a minimum of 24 hours prior to pre-weighing; although, a longer period of conditioning may be required. The weigh room laboratory temperature must be maintained between 20 and 23 °C, with no more than a ±2 °C change over the 24 period prior to weighing the filters. During transport from the weigh room to the sample location, there are no specific requirements for temperature control; however, the filters are located in their protective container and excessive heat/cold avoided. Temperature requirements for the sampling and post sampling periods are detailed in 40 CFR Part 50, Appendix L, CFR Part 50, Appendix L Section 7.4.10. These requirements state that the temperature of the filter cassette during sampler operation and in the period from the end of sampling to the time of sample recovery shall not exceed that of the ambient temperature by more than 5 °C for more than 30 minutes.

The specifics of temperature preservation requirements are clearly detailed in 40 CFR Part 50, Appendix L, CFR Part 50, Appendix L These requirements pertain to media both before and after the sample has been collected. Additionally, during the sample collection there are requirements for temperature control. The temperature requirements are detailed in Table 18.

# Table 18 Temperature Requirements

Item	Temperature Requirement	Reference
Weigh Room	20 – 23 °C	(40 CFR Part 50, Appendix L), Section 8.3.1
Pre-weighed Filter	±2 °C for 24 hours prior to weighing	(40 CFR Part 50, Appendix L), Section 8.3.2
Filter Temperature Control during	No more than 5°C above ambient	(40 CFR Part 50, Appendix L),
sampling and until recovery	temperature.	Section 7.4.10
Post Sample Transport so that final weight may be determined up to 30 days after end of sample period	4 °C or less	(40 CFR Part 50, Appendix L), Section 8.3.6

# 13.3 Laboratory QC and Corrective Action

A QC logbook and a database are maintained which contain QC data, including the microbalance calibration and maintenance information, routine internal QC checks of mass reference standards and laboratory and field filter blanks, and external QA audits. These computer records duplicate data recorded on laboratory data forms but will consolidate lab data so that long-term trends can be identified.

At the beginning of each weighing day, the laboratory balance operator performs a 5 minute internal auto calibration on the Mettler UMT2 microbalance. The operator next opens the GLIMS program (on the PC next to the balance) to weigh the calibration weight standards.

- Open GLIMS® and click the weigh icon.
- Click on the button labeled "Next Weigh is Cal."
- Open the black plastic box of Troemner weights on the weighing table & place the 100 mg weight on the balance.
- Once the weight is stable (the little square in the upper left disappears from the balance), click on "click here to read scale." Ensure the reading is close to 99.967 mg and is NOT zero. If the reading is zero, the standard must be weighed again. *Do not click on the* "invalidate last weigh result" button while weighing a standard. This will flag the weighing and they must be entered manually.
- Once the 100 mg weight passes this test, click on the "next weigh is Cal 2."
- The program is "ready for 200 mg weight."
- Repeat the weighing procedure with the 200 mg working standard.
- When finished with the working standards, weigh the 50 mg standard. This value does not go into GLIMS database instead enter it into the Balance Calibration Logbook. The weights of these three standards as well as the room conditions are also recorded in this logbook.

Lab filter blanks may now be weighted. Sharp forceps and the barcode scanner are used in the weighing procedure. Filters are weighed to the nearest microgram and are normally in the range of 120 and 160 milligrams.

Three laboratory filter blanks established for the current filter lot and three field filter blanks from the most recently completed field blank study are then weighed. After approximately every tenth filter weighing, the analyst re-weighs one working standard and zeros the microbalance. These weights are recorded in the GLIMS® database. If the working standard measurements differ from the certified values or the pre-sampling values by more than 3  $\mu$ g, the balance operator repeats the working standard measurements. If the blank measurements differ from the pre-sampling values by more than 15  $\mu$ g, the laboratory balance operator repeats the blank measurements. If the two measurements still disagree, the laboratory balance operator will:

- reweigh some or all of the previously weighed filters,
- recertify the working standard against the laboratory primary standard,
- conduct minor, non-invasive diagnostic and troubleshooting, and/or
- arrange to have the original vendor or an independent, authorized service technician trouble shoot or repair the Mettler UMT2 microbalance

If additional corrective action is required (see the tables below), filter weighing is delayed until corrective actions are satisfactorily implemented.

Table 19
Potential Problems/Corrective Action for Laboratory Support Equipment

System	Item	Problem	Action	Notification
Weigh Room	Humidity	Out of Specification	Check HVAC system	Lab Manager
Weigh Room	Temperature	Out of Specification	Check HVAC system	Lab Manager
Balance	Internal Calibration	Unstable	Redo & check working standards	Lab Manager
Balance	Zero	Unstable	Redo and check for drafts, sealed draft guard	Lab Manager
Balance	Working Standards	Out of Specification	Check balance with Primary standards	Lab Manager
Balance	Filter Weighing	Unstable	Check Lab Blank Filters	Document in Logbook

# 13.4 Filter Sample Contamination Prevention, Preservation, and Holding Time Requirements

This section details the requirements needed to prevent and protect the filter sample from contamination, the volume of air to be sampled, temperature preservation requirements, and the permissible holding times to ensure against degradation of sample integrity.

# 13.4.1 Sample Contamination Prevention

Filters are equilibrated/conditioned and stored in the same room where they are weighed. Powder free gloves are worn while handling filters and filters are only touched with the use of smooth non-serrated forceps. Upon determination of its pre-sampling weight, each filter is placed in a protective petri dish and then placed in a cassette. Petri dishes are labeled with bar coded numbers (site number and sample run date). Once a filter cassette is taken outside of the weigh room, it will never be opened as damage may result to the 46.2 mm Teflon filter.

# 13.4.2 Sample Volume

The required volume of sampled air is specified in 40 CFR Part 50, Appendix L. For a sample flow rate set at 16.67 l/min, a 24-hour sampling duration yields a total volume of 24 cubic meters of air.

# 13.4.3 Holding Times

Permissible holding times for the PM<sub>2.5</sub> sample is detailed in both 40 CFR Part 50, Appendix L and in Section 2.12 of the EPA Quality Assurance Handbook for Air Measurement Systems, Volume II Ambient Air Specific Methods. A summary of these holding times is provided in Table 16.

# 14.0 Quality Control Requirements

Quality control involves two distinct and interrelated functions. The first function is the control of the measurement process through broad quality assurance activities, such as establishing policies and procedures, developing data quality objectives, assigning roles and responsibilities, conducting oversight and reviews, and implementing corrective actions. The second function is the control of the measurement process through the implementation of specific quality control procedures, such as audits, calibrations, checks, replicates, and routine self-assessments.

Quality Control (QC) is the overall system of technical activities that measures the attributes and performance of the monitoring network. Table 20 lists QC activities employed by IDEM to evaluate and control data quality for the PM<sub>2.5</sub> network.

Table 20 Field QC Checks

Requirement	Requirement Frequency Acceptance Criter		CFR Reference	2.12 Reference	Information Provided	
Calibration Standards						
Flow Rate Transfer Std. 1/yr		±2% of NIST-traceable Std.	Part 50, App.L Sec 9.1, 9.2	Sec. 6.3	Certification of Traceability	
Field Thermometer	1/yr	±0.1 °C resolution	not described	Sec 4.2 and 8.3	Certification of Traceability	
		±0.5 °C accuracy	not described	"		
Field Barometer	1/yr	±1 mmHg resolution	not described	"	Certification of Traceability	
	-	±5 mmHg accuracy	not described	"		
Calibration/Verification						
Flow Rate (FR) Calibration	If multi-point failure	±2% of transfer standard	Part 50, App.L, Sec 9.2	Sec 6.3 and 6.6	Calibration drift and memory effects	
FR multi-point verification	1/yr	±2% of transfer standard	Part 50, App.L, Sec 9.2.5	Sec 8.3	Calibration drift and memory effects	
One point FR verification	1/4 weeks	±4% of transfer standard		Sec 8.3	Calibration drift and memory effects	
External Leak Check	every 5 sampling events	80 ml/min	Part 50, App.L, Sec 7.4	Sec. 8.3	Sampler function	
Internal Leak Check	every 5 sampling events	80 ml/min	"	Sec. 8.3	Sampler function	
Temperature Calibration	If multi-point failure	±2% of standard	Part 50, App.L, Sec 9.3	Sec. 6.4	Calibration drift and memory effects	
Temp multi-point verification	on installation, then 1/yr	±2 °C of standard	Part 50, App.L, Sec 9.3	Sec. 6.4 and 8.2	Calibration drift and memory effects	
One- point temp Verification	1/4 weeks	±4 °C of standard	standard "		Calibration drift and memory effects	
Pressure Calibration	on installation, then 1/yr 1/4	±10 mmHg	44	Sec. 6.5	Calibration drift and memory effects	
Pressure Verification	weeks	±10 mmHg	"	Sec. 8.2	Calibration drift and memory effects	
Clock/timer Verification	1/4 weeks	1 min/mo	Part 50, App.L, Sec 7.4	not described	Verification of to assure proper function	
Blanks						
Field Blanks	See 2.12 reference	±30 μg	Part 50, App.L Sec 8.2	Sec. 7.10	Measurement system contamination	
Precision Checks						
Collocated samples	Collocated samples every 6 days		Part 58, App.A, Sec 3.5, 5.5	Sec. 10.3	Measurement system precision	
Accuracy						
Flow rate audit	1/3mo (manual)	±4% of transfer standard	Part 58, App A, Sec 3.5.1	Sec. 8.1	Instrument bias/accuracy	
External Leak Check	External Leak Check 4/yr		not described	"	Sampler function	
Internal Leak Check	4/yr	< 80 ml/min	not described	"	Sampler function	
Temperature Check	Temperature Check 4/yr		not described	"	Calibration drift and memory effects	
Pressure Check	4/yr (?)	±10 mmHg		"	Calibration drift and memory effects	
Audits (external assessments)						
FRM Performance evaluation	25% of sites 4/yr	±10%	Part 58, App A, Sec 3.5.3	Sec 10.3	Measurement system bias	
Flow rate audit	1/yr	±4% of audit standard	not described	Sec 10.2	External verification bias/accuracy	
External Leak Check 1/yr		< 80 ml/min	not described		Sampler function	
Internal Leak Check 1/yr		< 80 ml/min	not described		Sampler function	
Temperature Audit 1/yr		±2 °C	not described		Calibration drift and memory effects	
Pressure Audit	1/yr	±10 mmHg	not described		Calibration drift and memory effects	

# Table 21 Laboratory QC

Requirement Frequency		Acceptance Criteria	QA Guidance Document 2.12 Reference	Information Provided	
Blanks					
Lot Blanks	3-lot	±15 μg difference	2.12 Sec. 7	Filter stabilization/	
Lab Blanks	3 per batch	±15 μg difference	Part 50, App.L Sec 8.2 2.12 Sec. 7.10	equilibrium Laboratory contamination	
Calib./Verification					
Balance Calibration	1/yr	Manufacturers spec.	2.12 sec 7.2	Verification of equipment	
Lab Temp. Calibration	3 mo	±2 °C	QAPP Sec. 13/16	operation	
Lab Humidity Calib.	3 mo	±2%	QAPP Sec. 13/16	Verification of equipment operation Verification of equipment operation	
Accuracy					
Balance Audit	1/year	±15 µg for unexposed filters	2.12 Sec 10.2	Laboratory technician operation	
Balance Check	beginning,		2.12 Sec. 7.8	1	
	every 10th samples, end	$\leq$ 3 µg		Balance accuracy/stability	
Calibration standards	•			, ,	
Working Mass Stds.	3-6 mo.	25 μg	2.12 Sec 4.3 and 7.3	Standards verification	
Primary Mass Stds.	1/yr	25 μg	m .	Primary standards verification	
Precision					
Duplicate weighing	1 per weighing session	±15 μg difference	2.12 Tab 7-1 QAPP Sec. 13/16	Weighing repeatability/filter stability	

# 14.1 Calibrations

Calibration is defined as the relationship between an instrument's output and the output of a known reference standard.  $PM_{2.5}$  calibration activities follow a two step process:

- Certification of a field calibration standard (transfer standard) against a laboratory reference standard
- Use of a field calibration standard to adjust an instrument under calibration

#### 14.2 Sample Blanks

Sample blanks determine contamination from five basic sources:

- 1. The sampling environment
- 2. The analysis environment
- 3. The reagents used in the analysis
- 4. The apparatus used
- 5. The operator/analysts performing the data operation

Three types of blanks are used in Indiana's PM<sub>2.5</sub> monitoring program.

- Lot Blanks: Lot blanks provide assurance of filter weight stability. Each shipment of filters from EPA is tested to determine the time filters take to weight stabilize or "de-gas". Upon receipt to the lab several filters are randomly selected from a lot and are placed in the weighing room (clean room). These filters are weighed every 24 hours until a stable weight is maintained (the re-weigh maintains a weight within ±15 μg). Once established, this time is used for each filter in that given lot.
- 2. Lab Blanks: Lab blanks provide an estimate of contamination in the clean room. One lab blank is randomly selected from a batch of 10-15 filters weighed clean filters. This blank remains in the clean room and is re-weighed when this batch of filters returns to the lab after sampling. Acceptance limit for a lab blank is a difference of 15 μg.
- 3. Field Blanks: Field blanks provide an estimate of total measurement system contamination. Laboratory blank data is compared to field blanks data in order to determine any contamination from field activities. Field blanks are used at the rate of 10-15% of the total samples. Acceptance limit for a field blank is a difference of 30 µg from initial to final weight.

Anytime any of the above blanks exceed the weight tolerance indicated, an investigation is conducted to determine the cause of the contamination.

#### 14.3 Precision Checks

#### **14.3.1** Collocated Samplers

Precision is the measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions. Precision for  $PM_{2.5}$  is estimated by the use of a duplicate or collocated sampler at a selected  $PM_{2.5}$  monitoring location in a measurement network. One sampler is designated as the reporting sampler and one sampler is designated as the collocated sampler. The collocated  $PM_{2.5}$  sampler must be maintained, operated, calibrated, and audited in the same manner as the reporting sampler. Precision is calculated from the difference in the concentrations from the reporting and collocated samplers over a calendar quarter. All collocated samplers operate on a 1 in 6 day (1/6) frequency. This allows for

approximately 15 data pairs (reporting & collocated concentrations) over each quarter for each site with collocated samplers. Estimates of network precision are made from three years of data.

Data is reported to the EPA AQS database for both the reporting and collocated sampler, regardless of concentration. However, CV is calculated only from data pairs (reporting and collocated concentrations) when both values are greater than 6 micrograms per cubic meter  $(\mu g/m^3)$ .

#### **14.3.2** Collocated Sampler Requirements

The duplicated sampler's inlet must be within 1 to 4 meters from the inlet of the reporting sampler's inlet and must be at least 2 meters from the inlet of any other sampler inlets such as high volume (TSP and/or PM<sub>10</sub>) samples. Four IDEM PM<sub>2.5</sub> sites have collocated samplers. This number meets the Federal Register/EPA requirement.

Two types of precision estimates are used in the PM<sub>2.5</sub> Program.

- 1. Collocated monitoring
- 2. Filter duplicates

The following formulas are used to calculate precision from reporting and collocated data pairs. These formulas are also stated in 40 CFR Part 50, Appendix L and 40 CFR Part 58 Appendix A.

Percent Difference for a Single Check  $(d_i)$ . The percentage difference,  $d_i$ , for each check is calculated by using Equation 1, where  $X_i$  represents the concentration produced from the primary sampler and  $Y_i$  represents the concentration reported for the duplicate sampler.

Equation 1: 
$$d_i = \frac{Y_i - X_i}{\frac{(Y_i + X_i)}{2}} \times 100$$

Coefficient of Variation (CV) for a Single Check ( $CV_i$ ). The coefficient of variation,  $CV_i$ , for each check is calculated by dividing the absolute value of the percentage difference,  $d_i$ , by the square root of two as shown in Equation 2.

Equation 2: 
$$CV_i = \frac{|d_i|}{\sqrt{2}}$$

Precision of a Single Sampler - Quarterly Basis  $(CV_{j,q})$ . For particulate sampler j, the individual coefficients of variation  $(CV_{j,q})$  during the quarter are pooled using Equation 3, where  $n_{j,q}$  is the number of pairs of measurements from collocated samplers during the quarter.

Equation 3: 
$$CV_{j,q} = \sqrt{\frac{\sum_{i=1}^{n_j} CV_i^2}{n_{j,q}}}$$

The 90 percent confidence limits for the single sampler's CV are calculated using Equations 4 and 5, where  $\chi^2_{0.05,df}$  and  $\chi^2_{0.95,df}$  are the 0.05 and 0.95 quantiles of the chi-square ( $\chi^2$ ) distribution with  $n_{i,a}$  degrees of freedom.

Equation 4: Lower Confidence Limit = 
$$CV_{j,q} \sqrt{\frac{n_{j,q}}{\chi^2_{0.95,n_{j,q}}}}$$

Equation 5: Upper Confidence Limit = 
$$CV_{j,q} \sqrt{\frac{n_{j,q}}{\chi^2_{0.05,n_{j,q}}}}$$

Precision of a Single Sampler Annual Basis - For particulate sampler j, the individual coefficients of variation,  $CV_i$ , produced during the calendar year are pooled using Equation 3, where  $n_j$  is the number of checks made during the calendar year. The 90 percent confidence limits for the single sampler's CV are calculated using Equations 4 and 5, where  $\chi^2_{0.05,df}$  and  $\chi^2_{0.95,df}$  are the 0.05 and 0.95 quantiles of the chi-square ( $\chi^2$ ) distribution with  $n_j$  degrees of freedom.

Corrective Action: Single Monitor - The precision data quality objective of 10% coefficient of variation (CV) is based upon the evaluation of three years of collocated precision data. CV values of greater than 10% may occur within that three year period. Single collocated pairs with values greater than 10% are flagged (FCS) and filters are re-weighed. If the CV remains between 10-20% the field technician is alerted to the problem and other operation solutions are investigated. If the CV is greater than 20% for both the initial and reweigh, all the primary sampler data is flagged (FCS) from the last precision check and corrective action is initiated. Paired CVs and percent differences are control charted to determine trends (Section 14.2).

Corrective Action: Quarter - Corrective action is usually initiated and imprecision rectified before a calendar quarter of data fails to meet the 10% CV limit. However, in the case where a quarter's CV is greater than 20% data for that monitor for that quarter is flagged. The EPA Regional Office is alerted of the issue and may be asked to help find a common solution.

Duplicate Laboratory Measurements - During laboratory pre-weighing and post-weighing sessions, a filter from a batch is selected for a second weighing. The acceptable limit for the difference between the first post-weight and the second post-weight is 15  $\mu$ g for clean filters and 30  $\mu$ g for exposed filters. If this limit is not met, the pair of values is flagged FLD. Failure may

be due to transcription errors, microbalance malfunction, or the samples have not reached equilibrium. Other QC checks (balance standards and lab blanks) will eliminate microbalance malfunction. If the duplicate does not meet the criterion, a second sample is selected and reweighed as a second duplicate check. If this second check fails the acceptance criterion and the possibility of balance malfunction and transcription errors have been eliminated, all samples in

the batch are equilibrated for an additional 24 hours and re-weighed. Corrective actions continue until duplicate weights for the batch meet acceptance criteria.

#### 14.4 Accuracy or Bias Checks

Accuracy is defined as the degree of agreement between an observed value and an accepted reference value. Four accuracy checks are used in the PM<sub>2.5</sub> program:

- 1. Collocated sampler
- 2. Flow rate audit
- 3. Balance check
- 4. Performance Evaluation Program (PEP Audit)

Collocated Samplers - Collocated samplers are primarily used for estimating precision; however, they also can be used to determine accuracy or bias. Equation 1 is used to determine percent difference so that bias may be calculated. Use of the FRM performance evaluation information (discussed below) in conjunction with collocation data is used to improve the data quality.

Corrective Action - The percent difference of the paired values is reviewed to determine trends. If it appears that there is a statistically significant bias (> 10% at the 90% confidence level) between the pairs, corrective action is initiated. The process includes eliminating uncertainties that may be occurring at filter handling, transport and laboratory stages, in order to determine that the bias is truly at the instrument. Corrective actions at the instrument include multi-point temperature, pressure, and flow rate checks as well as complete maintenance activities. Additional corrective action includes a request for vendor repairs or a request to Region 5 for a FRM performance evaluation.

Flow Rate Audit - IDEM conducts a flow rate audit on all samplers once each calendar quarter. The sampler's normal operating flow rate is measured with a certified flow transfer standard (FTS) audit device. The audit flow rate (true flow) is in actual conditions and the corresponding sampler's flow rate (observed flow) is indicated on its LCD display. The procedures used to calculate measurement uncertainty are described below.

Accuracy of a Single Sampler - Single Check (Quarterly) Basis  $(d_i)$  - The percentage difference  $(d_i)$  for a single flow rate audit i is calculated using Equation 6, where  $X_i$  represents the audit standard flow rate (true flow) and  $Y_i$  represents the sampler's flow rate (observed).

Equation 6: 
$$d_i = \frac{Y_i - X_i}{X_i} \times 100$$

Bias of a Single Sampler - Annual Basis  $(D_j)$  - For an individual particulate sampler j, the average  $(D_i)$  of the individual percentage differences  $(d_i)$  during the year is calculated using

Equation 7, where  $n_j$  is the number of individual percentage differences produced for sampler j during the year.

Equation 7: 
$$D_j = \frac{1}{n_i} \sum_{i=1}^{n_j} d_i$$

Bias for Each EPA Federal Reference and Equivalent Method Designation employed by IDEM - Quarterly Basis  $(D_{k,q})$  - For method designation k used by the reporting organization, quarter q's single sampler percentage differences  $(d_i)$  are averaged using Equation 8, where  $n_{k,q}$  is the number of individual percentage differences produced for method designation k in quarter q.

**Equation 8:** 
$$D_{k,q} = \frac{1}{n_{k,q}} \sum_{i=1}^{n_{k,q}} d_i$$

Corrective Action - The single sampler accuracy acceptable limit is  $\pm 4\%$  (pass if  $< \pm 4\%$  and fail if  $\geq \pm 4\%$ ). If the sampler fails an audit, an external/internal leak check is performed. Temperature and pressure sensors are also audited and then the flow audit is repeated. If the audit is still unacceptable, a multi-point calibration followed by a one-point verification is performed. Routinely, data back to the last passing audit or verification is flagged and reviewed to determine validity (see Section 23). A verification is an audit performed by the site operator once each month (see Section 16).

Balance Checks - Balance checks are routine verifications using working standard weights (100 and 200 mg) to ensure that the balance is within acceptance criteria throughout the pre- and post-sampling weighing sessions. IDEM uses Troemner Class 1 weights for its primary and secondary (working) standards. Working standards are used at the beginning and end of each batch of weighed samples. In addition, one standard is selected for a check comparison after every 10 filters.

Balance Check Evaluation - The following formula is used to evaluate the balance checks.

Difference for a single check  $(d_y)$  - The difference,  $d_y$ , for each check is calculated using Equation 9, where X represents the certified mass weight and Y represents the reported weight.

Equation 9: 
$$d_y = Y - X$$

Corrective Action - The difference between the reported weight and the certified weight must be  $\leq 3~\mu g$ . Since this is the first check before any pre- or post-sampling weighings, if the acceptance criterion is not met, corrective action is initiated. Corrective action may be as simple as allowing the balance to perform internal calibrations or to sufficiently warm-up, which may require checking the balance weights a number of times. If the acceptance criteria are still not met, the laboratory technician is required to verify the working standards against the primary standards.

Finally, if it is established that the balance does not meet acceptance criteria for both the working and primary standards, and other trouble shooting techniques fail, a *Mettler* service technician (see Section 15) is called to perform corrective action.

If the balance check fails acceptance criteria during a run, the 10 filters weighed prior to the failure are re-weighed. If the balance check continues to fail, troubleshooting procedures are initiated. The values of the 10 sample filters weighed prior to the failure are recorded but will remain with the un-weighed samples in the batch to be re-weighed when the balance meets the acceptance criteria.

Performance Evaluation Program (PEP) - The Federal Reference Method (FRM) Performance Evaluation Program is a national quality assurance activity that is used to evaluate measurement system bias of all PM<sub>2.5</sub> monitoring networks. The strategy is to collocate a portable FRM PM<sub>2.5</sub> PEP sampling instrument with an established air monitoring site, operate both monitors in the same manner, and then compare the concentrations of the PEP sampler with the established network sampler. The EPA has implemented this program. The EPA Region V office oversees the Indiana samplers and a contractor operates the PEP sampler. The contractor informs IDEM when an evaluation is scheduled, sets up one or more samplers at one or more sites, then collects sample(s) on the normal sampling schedule. PEP sampler filters are sent to a national laboratory in Region 10 for gravimetric analysis. EPA evaluates this data by using the IDEM concentrations reported to the AQS database and the data from the Region 10 analysis. This performance evaluation is an estimate of the uncertainty of the agency's measurement system but may also be used to compare different models and brands of samplers. Biases may be attributed to sample handling, transportation and laboratory activities as well as to the instrument.

Corrective Action - EPA notifies IDEM of the evaluation results. The bias acceptance limit for the data comparison is  $\pm 10\%$ . If it appears that there is a bias, corrective action is initiated. Corrective actions usually begin with evaluating the data collection procedures and then the laboratory procedures. EPA Region V may conduct additional PEP audits to provide additional data to troubleshoot the process.

#### 14.5 Sample Batches - QC Sample Distribution

Samples are organized into batches to aid in the QC weighing activities. A batch of samples consists of all routine and QC samples collected in a one week sample period.

Table 22 Sample Batch

Sample	Number
32 sites 1/3 day sampling	99
4 sites 1/6 day sampling	3
lab blanks	5
field blanks	5
11 collocated monitor	11
Total	131

#### 15.0 Sampler Maintenance

In general, a routine maintenance program is designed to eliminate data loss due to preventable failures of monitoring equipment. The Ambient Air Monitoring Section has developed a maintenance schedule for each sampler in the program. Logbooks for each sampler are kept on site.

#### 15.1 Routine Maintenance Procedures

- 1. Filter cassettes Inspect filter cassettes for contamination after every use. Wipe with a clean cloth as required.
- 2. WINS Impactor clean or change the WINS impactor after every five sampling days. It is the policy of the Indiana Department of Environmental Management to perform WINS maintenance in a separate controlled environment (laboratory, field office etc.) whenever possible. Prepared WINS impactors are transported to the site in an upright position to avoid spilling the impactor oil. Refer to the Ambient Section's SOP for detailed instructions on impactor maintenance.
- 3. Internal Leak Check Perform an internal leak check according to the Ambient SOP after every five sampling episodes or as indicated by sampler diagnostics.
- 4. External Leak Check Perform an external leak check according to the Ambient SOP after every five sampling episodes or as indicated by sampler diagnostics.
- 5. 1<sup>st</sup> Stage Inlet Monthly, disassemble the R&P Partisol-Plus Air Sampler sample inlet and clean with a soft brush or cloth.
- 6. In-line Filter Every six months replace the in-line filter according to the R&P Partisol-Plus Air Sampler operating manual or IDEM SOPs.
- 7. V Seals Check V seals once each year and replace if necessary.

- 8. Air Screens Clean the samplers air screen (located under the sampler rain hoods) every six months.
- 9. Battery Voltage Check the voltage of the batteries on the main computer board in the electrons compartment every six months.

Table 23
Inspection of Field Items

Item	Item Inspection Frequency		Action if Item Fails Inspection	Documentation Requirement	
Sample downtube	Every site visit	Visible particulate	Clean with a clean dry cloth.	Document in logbook.	
WINS Impactor well	Every site visit	"Cone" shape of particulate on impactor well	Replace impactor well (including new impactor oil).	Document in logbook.	
Rain collector	Every site visit	>1/3 full	Empty.	Document in logbook.	
O-rings	Every site visit	Any damage	Replace.	Document in logbook.	
Filter Cassettes	After each sample run	each sample Visible Check do particulate WINS in		Document in logbook.	
Cassette Seals	Each sample	Clean and smooth	Clean with a clean dry cloth, or replace as needed.	Document when replaced.	
In-line filter	Every 6 months	Loaded particulate	Replace.	Document in logbook.	
Battery	Battery Every 6 months Decrease in voltage		Replace.	Document in logbook.	

Sample recovery must be performed within 168 hours from the end of the sample period. The table below illustrates set-up, run, and recovery dates based on sample frequency requirements of a 1 in 3 day sampling frequency.

Table 24
Sample Set-up, Run, and Recovery Dates

Sample Frequency	Sampler Type	Sunday	Monday	Tuesday	Wednesday	Thursday	Friday	Saturday
1 in 3	Multiple	Sample			Sample	Recovery &		Sample
Week 1	Day	Day 1			Day 2	Set-up		Day 3
1 in 3	Multiple			Sample	Recovery &		Sample	
Week 2	Day			Day 4	Set-up		Day 5	
1 in 3	Multiple		Sample	Recovery &		Sample		
Week 3	Day		Day 6	Set-up		Day 7		
1 in 3	Multiple	Sample	Recovery		Sample	Recovery &		Sample
Week 4	Day	Day 8	& Set-up		Day 9	Set-up		Day 10
1 in 3	Multiple			Sample	Recovery &		Sample	
Week 5	Day			Day 11	Set-up		Day 12	
1 in 3	Multiple		Sample	Recovery &		Sample		
Week 6	Day		Day 13	Set-up		Day 14		

# 16.0 PM<sub>2.5</sub> Sampler Calibration

The R&P PM<sub>2.5</sub> sampler requires four calibrations:

- 1. Flow rate controller
- 2. Ambient temperature sensor
- 3. Filter temperature sensor
- 4. Barometric pressure sensor

#### **16.1 Flow Rate Comparison**

Regulations in 40 CFR Part 50, Appendix L, require that  $PM_{2.5}$  sampler flow rates to be calibrated in actual volumetric flow rate at ambient conditions of temperature and pressure ( $Q_a$ ).  $PM_{2.5}$  concentrations are calculated based on the actual volume at ambient conditions of temperature and pressure. Flow Transfer Standards (FTS) that are calibrated at standard conditions,  $Q_{std}$ , are converted to  $Q_a$ .

- Q<sub>a</sub> Actual volumetric air flow rates are measured at ambient conditions of temperature and pressure. Units of actual volumetric flow rates are expressed in l/min or a l/min and in m³/min or am³/min. Inlet design flow rates are given in actual volumetric flow units.
- Q<sub>std</sub> Flow rates that have been corrected to standard conditions of temperature and pressure of 25 °C (298 °K) and 760 mmHg (101 kPa). Units of standard flow are expressed in stdl/min or stdm<sup>3</sup>/min. Standard volume flow rates are equivalent to mass flow rate units.

Flow rate units may be converted as follows:

$$Q_{std} = Q_a(P_a/P_{std})(T_{std}/T_a)$$
 
$$Q_a = Q_{std}(P_{std}/P_a)(T_a/T_{std})$$

Where:

 $Q_{std}$  = standard volume flow rate, m³/min  $Q_a$  = actual volume flow rate, m³/min  $P_a$  = ambient barometric pressure, mmHg  $P_{std}$  = standard barometric pressure, 760 mmHg  $T_{std}$  = standard temperature, 298 °K (25 °C + 273)  $T_a$  = ambient temperature, °K (°C + 273)

A flow rate measured in actual volumetric units  $(Q_a)$  is always associated with the temperature and pressure of the gas. When temperature or pressure changes volumetric flow rate also changes even though the mass flow rate of the gas remains the same. Therefore, when the flow rate is measured at different points in the sampler, the volumetric flow rate observed is different if either the temperature or the pressure is different. For example, when a flow calibration device

is connected to the sampler inlet, the pressure of the airflow measured by the flow calibration standard is the ambient barometric pressure. The pressure of the airflow measured by the sampler's flow measurement system; however, is somewhat lower than the ambient pressure because all flow calibration devices cause some pressure drop.

If this pressure drop is negligible, there is not a problem; the pressure can be considered the same for both measurement systems. But if the pressure drop is significant, then the volumetric flow rate measured by the two systems will be different, and this difference should be taken into account when comparing them. Sample flow rate measurement systems may or may not automatically correct for this pressure change. Staff should consult the sampler's operating manual. Use the following formula for a manual correction:

$$Q_1 = Q_2(P_2/P_1)(T_1/T_2)$$

Where:

 $Q_1$  = actual volume flow rate am<sup>3</sup>/min, at pressure of first point, am<sup>3</sup>/min

 $Q_2$  = actual volume flow rate, am<sup>3</sup>/min, at second point am<sup>3</sup>/min

 $P_1$  = pressure at first point mmHg

 $P_2$  = pressure at second point mmHg

 $T_1$  = temperature at first point K (°C +273)

 $T_2$  = temperature at second point K (°C +273)

#### 16.2 Flow Rate Calibration

A detailed calibration procedure for the R&P Partisol-Plus Air Sampler is contained in the operations manual 40 CFR Part 50, Appendix L and CFR Part 53. The following are relevant points about flow rate calibration.

- A three-point calibration and single point verification of each sampler's flow rate is routinely performed. Calibrations are performed with a flow transfer standard (FTS) that has been certified against a NIST-traceable standard. This NIST traceability is required by 40 CFR Part 50, Appendix L, CFR Part 50, Appendix L, Part 58.
- R&P Partisol-Plus Air Samplers can accommodate various types of flow measurement devices. The specific calibration standard and procedure used for the calibration or verification of the sampler's flow rate varies depending on the type of flow rate measurement system employed 40 CFR Part 50, Appendix L. Consult the sampler operations manual for the recommended standard type and its specific calibration procedure.
- Calibration of the sampler's flow rate measurement system is reported in units of actual volumetric flow rate.
- The sampler flow rate measurement system is calibrated or verified by installing a filter in the

- holder. This filter provides the necessary flow resistance for a calibration and should never be used for sample collection. The operator should verify that no leaks exist between the flow rate standard and the sampler (40 CFR Part 50, Appendix L, Sec. 9.2.4).
- IDEM routinely uses a Chinook Engineering Streamline® FTS to measure flow rate.
- Monthly verification of the sampler's flow rate consists of one-point check of the sampler's operational flow rate (40 CFR Part 50, Appendix L, Sec. 9.2.4). A one-point verification may be used as a calibration provided that a three-point calibration is performed on initial sampler installation and at least once per year. A three-point calibration is performed if the one-point verification indicates that the sampler's flow rate differs by ±4 percent or more from the flow rate measured by the certified FTS device.
- A multi-point calibration of the sampler's flow rate consists of three flow rate measurements evenly spaced within the range of ±10 percent of the sampler's operational flow rate (40 CFR Part 50, Appendix L, Sec. 9.2.4). The sampler is required to have the capability to adjust the flow rate over the ±10 percent range (40 CFR Part 50, Appendix L, Sec.7.2.2). The R&P Partisol-Plus Sampler operations manual provides additional guidelines on flow rate adjustments.
- Following a calibration or verification, the FTS is disconnected from the sampler and the sampler's inlet is reinstalled. The calibration filter remains in place. Then the sampler's normal operating flow rate is read from the LCD display on the sampler. If the sampler flow rate differs by ±2 percent or more from the required operational value of 16.67 l/min, the sampler flow rate must be adjusted to the specified flow rate. The calibration filter is then removed and the sampler is returned to normal "ready to sample" mode.

A short calibration procedure for the R&P Partisol-Plus Sampler is as follows:

- 1. On the sampler's keyboard and LCD screen, access the Service Menu and select FlowCal.
- 2. Enter or confirm the slope and intercept constants of the FTS.
- 3. Remove the inlet head from the down tube.
- 4. Remove either the sample filter or spacer and install a calibration filter.
- 5. Attach the FTS to the sample tube.
- 6. On the Flow Calibration screen, press <Edit> and enter the desired minimum and maximum calibration flow rates as well as the number of calibration points desired, press <Enter>.
- 7. Initiate the flow calibration.
- 8. Allow the flow to stabilize for each calibration point. Enter either the pressure drop (inches of water) from the FTS or the flow (volumetric l/min) from a flow meter. The sampler will proceed to the next flow rate point after the user has properly entered each measured value.

- 9. The sampler adjusts the Offset and Span values in the calibration screen once it performs measurements at all the flow rate points.
- 10. Restore the sampler to its pre-calibration state by removing the flow metering devices and reinstalling the inlet head onto the external sample tube

# 16.3 Temperature Sensor Calibration

# **16.3.1 Temperature Standards**

The EPA *Quality Assurance Handbook Volume IV (EPA1995), Section 4.3.5.1*, provides information on calibration equipment and methods for assessing response characteristics of temperature sensors. The ambient air and filter temperature sensors of R&P Partisol-Plus Air Sampler are required to have a resolution of 0.1 °C and an accuracy of ±2 °C over the range of -30 to 45 °C. The handbook describes how to prepare three stable thermal mass assemblies (insulated vacuum bottles containing pure water and ice) whose temperatures can be determined to about 0.1 °C.

# 16.3.2 NIST Traceablility and Certification of Temperature Transfer Standards

Each temperature standard used for temperature calibration is traceable to a NIST primary standard. These transfer standards are verified or certified at least annually by the IDEM Quality Assurance Certification Laboratory.

# **16.3.3** Temperature Sensor Calibration Procedure

Both the ambient air and filter temperature sensors are calibrated once per year. The ambient air sensor is located inside the shielded fixture on the outside of the  $PM_{2.5}$  sampler. The filter compartment temperature sensor is located inside the sampler near the filter holder assembly. One-point calibrations are performed in the field. Sampler-specific procedures and instructions are found in the operator's manual for the R&P Partisol-Plus Sampler.

#### 16.4 Pressure Sensor Calibration

The R&P Partisol-Plus Air Sampler uses a barometric pressure sensor to correct the actual sampling flow rate to the design value of 16.67 l/min. The sampler's operations manual provides information on how to make adjustments to calibrate the pressure sensor.

## **16.4.1** General Requirements

- 1. The barometric pressure sensor of the R&P sampler has a range of 600 to 800 mmHg with a resolution of 5 mmHg and an accuracy of  $\pm 10$  mmHg.
- 2. The sensor is calibrated by comparing it to a secondary standard that is traceable to a primary standard.

- 3. The Fortis-type mercury barometer is used as a laboratory primary standard. The primary standard is used to certify the accuracy of aneroid barometers (field standards). Aneroid barometers are less accurate than the Fortis type, but can be transported with less risk to the reliability of its measurements and they present no hazard to personnel from mercury spills. An alternative to an aneroid barometer as a field standard is a Digital Hand-Held Barometer (DHHB). The DHHB is a small, light instrument that can accurately measure and display atmospheric pressure and pressure altitude. A microprocessor computes instantaneous readings of ambient pressure and pressure altitude from the output of an accurate pressure sensor. A standard 9-volt battery powers the unit.
- 4. As with all associated instrumentation, care is taken to minimize violent mechanical shock. Should any barometer be subjected to sudden changes in pressure (air transport etc.) it is recalibrated. Locate the instrument so as to avoid direct sunlight, drafts or indiscriminate temperature changes, and vibration.

# 16.4.2 Aneroid Barometer and Digital Barometer Calibration

# Aneroid Barometer

- 1. Always use and read an aneroid barometer when it is in the same position (vertical or horizontal) as when it was calibrated. Place the portable aneroid barometer next to the Quality Assurance laboratory's primary standard (Fortis type barometer) to begin the calibration.
- 2. Immediately before reading the scale on the aneroid barometer, lightly tap the instrument casing to free up the indicating needle (pointer) from any bearing drag.
- 3. Read the aneroid barometer to the nearest 1mmHg. Compare the aneroid barometer (transfer standard) to the laboratory's primary standard. If necessary, adjust the reading on the portable barometer to match those from the primary standard.

# <u>Digital Hand-Held Barometer (DHHB) – Novalynx Model 230-AIR-HB-1A & 2A</u>

- 1. Enter the Barometer Operating Mode by pressing the MODE key until the PRESSURE annuciator is illuminated in the upper left of the display.
- 2. Press an arrow key until the desired pressure units are displayed.
- 3. Make a careful comparison of the barometric pressure standard reading to the DHHB reading, averaging readings over one minute.
- 4. Determine the adjustment by subtracting the DHHB value from the barometric pressure standard.
- 5. The resulting difference (retaining the algebraic sign) is the adjustment value.
- 6. Press and hold down the SET/ZERO key then press.
- 7. While holding the SET/ZERO key, press and hold down the MODE key. The display will show the letters "CAL" for one second and then the current pressure adjustment is displayed. The DHHP is shipped with a pressure adjustment of 0.0.
- 8. Holding the SET/ZERO and MODE keys depressed, press the up arrow key to increase this adjustment, or the down arrow key to decrease the adjustment. The pressure adjustment must

be in the range of -1.28 mb to +1.27 mb. If the measured adjustment is outside this range, the accuracy of the barometric pressure standard should be verified. If the barometric pressure standard is correct, return the DHHB to the manufacturer for recalibration.

1.0 inch of mercury	33.86 millibars (mb)
1.0 millimeter of mercury	1.33 millibars
1.0 inch of water	2.49 millibars

#### 16.4.3 Pressure Sensor Calibration Procedure

Using a certified aneroid or digital barometer as a pressure transfer standard, determine the ambient station pressure. Following the directions in the R&P Partisol-Plus Sampler operations manual, enter the ambient pressure in mmHg. The R&P Partisol-Plus Sampler automatically adjusts the corresponding offset based upon this input. Record all readings in the sampler logbook.

# **16.5** Sampler Component Calibration Frequency

- 1. A flow rate multi-point calibration is performed annually or when a monthly one-point flow rate verification does not meet the requirements.
- 2. A temperature sensor multi-point calibration is performed annually or when a monthly one-point temperature check fails to meet the accuracy requirement of  $\pm 2$  °C from standard.
- 3. A pressure sensor calibration is performed annually or when a monthly pressure sensor check fails to meet the accuracy requirements of  $\pm 10$  mmHg from standard. (Section 5.5.5).

# 17.0 Inspection/Acceptance for Supplies and Consumables

# 17.1 Purpose

This section establishes and documents a system for inspecting and accepting all supplies and consumables that may directly or indirectly affect the quality of Indiana's PM<sub>2.5</sub> Monitoring Program. The Indiana network relies on various supplies and consumables that are critical to its operation. Documented inspections and acceptance criteria assures consistency of the supplies.

# 17.2 Critical Supplies and Consumables

The following table lists the needed supplies and includes items for the weigh room laboratory and the field.

# Table 25 Critical Supplies

Area	Item	Description	Vendor	Model Number
Sampler	Impactor Oil	Tetramethyltetraphenyl- trisiloxane (30 ml)	Fisher Scientific	Cat # 01-185-2A
Sampler	37 mm Glass Fiber Filter	For use in impactor well	Fisher Scientific	Cat #-0-730-8A
Sampler	Rain Collector	Glass	R & P	32-000625
Sampler	O-Rings	The filter cassette O-rings.	R & P	Upper: 22-002182 Lower: 22-004276
Sampler	In-line Filter	Downstream of sample & upstream of sample pump.	R & P	32-002643
Sampler	Battery	Internal Sampler Battery.	Rayovac	AA
Sampler	Fuses	In sampler	R & P	59-005129
Filter	Filters	46.2 mm teflon	EPA	
Filter	Petri Dish	47 mm with securing ring.	Falcon	60 x 15 mm dishes Falcon 1007
Filter	Filter Cassettes (single)	As per CFR design	R & P	59-004648
Filter	Filter Cassette Holder, Protective Containers	For securing cassette	R & P	20-004997
Filter	Sequential Sampler Cassette Holder	For use with R&P Partisol- Plus Air Sampler model 2025	Same as Single	
Filter	Filter Handling Containers	Transport to & from field	Polyfoam Packers Corp.	458
Weigh Room	Static Wipes			Texwipe Texstat 100, TX926
Weigh Room	Static Control Strips	Polonium 500°C <sub>i</sub>	NRD, Inc.	3" Strips are 2U500 1" Strips are 1U400
Weigh Room	Air Filters	High Efficiency	EPA Filters	
All	Powder Free Antistatic Gloves	Vinyl, Class M4.5	Phoenix Medical Technology	PV602
All	Low-lint wipes	4.5" x 8.5" Wipes	Cole Palmer	Cat # E-33670-00

# 17.3 Acceptance Criteria

Acceptance criteria must be consistent with overall project technical and quality criteria. Some of the acceptance criteria are specifically detailed in 40 CFR Part 50, Appendix L. Other acceptance criteria such as observation of damage due to shipping can only be performed once the equipment has arrived on site.

Table 26 lists the acceptance test and limits for procurement of supplies and consumables used in the Indiana PM<sub>2.5</sub> network:

# Table 26 Acceptance Criteria for Supplies

Equipment	Acceptance Criteria	Action if Requirements not met
Impactor Oil	Is oil identified as Tetramethyltetraphenyl-trisiloxane	Return
37 mm Glass Fiber Filter	Filters of the correct size and quality	Return
Rain Collector	Not broken	Call Vendor, will not return
O-Rings	Of the correct size	Return
Battery	Correct size and voltage	Return
Fuses	Correct size and specification	Return
Floppy Disks	Undamaged and pre-formatted	Return
Filters, 46.2 mm Teflon	Tested & Accepted by EPA with documentation of acceptance in package. Should meet visual inspection & preweight (110-160mg) criteria	Call David Lutz, U.S. EPA (919) 541-5476
Petri-dish	Clean and appropriately sized for 46.2 mm filters	Return
Filter Cassettes (single)	Of the correct type and make	Return
Filter Cassette Holder, Protective Containers	Of correct size so that filter cassettes won't move around that could lead to dislodging particulate	Return
Sequential Sampler Cassette Holder	Of the correct type for use with the sequential sampler model	Return
Filter Handling Containers	Clean	Clean
Anti-Static Solution	Of the correct type	Return
Static Control Strips	Manufactured within past 3 months and between 400 and 500°C <sub>i</sub> of Polonium	Call vendor
Air Filters	Of the size and quality specified	Return
Powder Free Antistatic Gloves	Of the size and quality specified	Return
Cleaning Wipes	Of the quality specified	Return

# 17.4 Tracking and Quality Verification of Supplies and Consumables

Tracking and quality verification of supplies and consumables have two main components. The first is the need of the end user of the supply or consumable to have an item of the required quality. The second need is for the IDEM purchasing department to accurately track goods received so that payment or credit of invoices can be approved. In order to address these two issues, the following outlines proper tracking and documentation procedures:

- 1. Perform inspection of packages as they are received. Note any problems with a shipment such as crushed box or wet cardboard.
- 2. Open packages, inspect, and check contents against the packing slip.
- 3. Compare supplies/consumables to the acceptance criteria in Table 26.

- 4. If there is a problem with the equipment/supply, notify the appropriate staff (ambient, analytical, quality assurance). The vendor may also need to be contacted.
- 5. If the equipment/supplies appear to be complete and in good condition, forward paper work to the purchasing department so that payment can be made in a timely manner.
- 6. Notify appropriate personnel that the equipment/supplies are available.
- 7. Store equipment/supplies in appropriate areas.

All changes in supplies, consumables, and equipment used throughout the Indiana's  $PM_{2.5}$  program must be documented. All relevant information such as model number, lot number, and serial number is included in this documentation.

# 18.0 Data Acquisition Requirements

This section addresses non-monitoring data that is associated with the PM<sub>2.5</sub> Ambient Air Quality Monitoring Program. This includes both outside data and historical monitoring data. IDEM uses non-monitoring data and historical monitoring data in a variety of ways. Use of information that fails to meet the necessary Data Quality Objectives (DQOs) for the PM<sub>2.5</sub> Ambient Air Quality Monitoring Program can lead to erroneous trend reports and regulatory decision errors. The policies and procedures described in this section apply both to data acquired from the Indiana Department of Environmental Management's monitoring program and to information previously acquired and/or acquired from outside sources.

# 18.1 Acquisition of Non-Direct Measurement Data

The PM<sub>2.5</sub> Ambient Air Quality Monitoring Program relies on data that is generated through field and laboratory operations; however, other significant data is obtained from sources outside the IDEM or from historical records.

## Chemical and Physical Properties Data

Physical and chemical properties data and conversion constants are often required in the processing of raw data into reporting units. This type of information that has not already been specified in the monitoring regulations is obtained from nationally and internationally recognized sources. Other data sources may be used with approval of the Air Monitoring QA Section Chief. The following sources may be used in the PM<sub>2.5</sub> Ambient Air Quality Monitoring Program without prior approval:

- National Institute of Standards and Technology (NIST)
- ISO, IUPAC, ANSI, and other widely-recognized national and international standards organizations
- USEPA
- The current edition of certain standard handbooks may be used without prior approval of the QA Section Chief. Two that are relevant to the fine particulate monitoring program are CRC Press' *Handbook of Chemistry and Physics* and *Lange's Handbook*.

# Sampler Operation and Manufacturer's Literature

Another important source of information needed for sampler operation is the manufacturer's literature. Operations manuals and user manuals frequently provide numerical information and equations pertaining to specific equipment. Whenever possible, the field operators should compare physical and chemical constants in the operators manuals to those given in the sources listed above. If discrepancies are found, the Rupprecht & Patashnick Company is contacted. If a change is indicated, IDEM contacts the Region 5.

# Geographic Location

Another type of data that will commonly be used in conjunction with the PM<sub>2.5</sub> Ambient Air Quality Monitoring Program is geographic information. For the current sites, IDEM uses a global positioning system (GPS) that meets EPA Locational Data Policy of 25 meters accuracy. USGS maps were used as the primary means for locating and siting stations in the existing network.

# Historical Monitoring Information of the IDEM

Ambient air monitoring stations have been operated by an Indiana state agency since early 1970's. Since 1986, the Indiana Department of Environmental Management has operated the stations. Prior to 1986, monitoring was conducted by the Indiana State Board of Health, Air Pollution Control Division. Historical monitoring data and summary information derived from this past data may be used in conjunction with current monitoring results to calculate and report trends in pollutant concentrations. If calculating historical trends, IDEM first verifies that historical data are fully comparable to current monitoring data. If different methodologies were used to gather the historical data, the biases and other inaccuracies are described in trends reports based on that data. Direct comparisons of PM<sub>2.5</sub> with historical TSP or PM<sub>10</sub> data will not be reported or used to estimate trends. Trends reports comparing PM<sub>2.5</sub> data with historical particulate data are approved by the QA Section Chief prior to release.

## External Monitoring Databases

It is the policy of the IDEM that no data obtained from the Internet or databases from outside organizations shall be used in creating reportable data or published reports without approval of the Air Monitoring Branch Chief. This policy is intended to ensure the use of high quality data in all IDEM Air Monitoring publications.

Data from the EPA AQS database may be used in published reports. Care is taken in reviewing/using any data that contains flags or data qualifiers. If data is flagged, such data will not be used unless it is clear that the data still meets critical QA/QC requirements.

#### U.S. Weather Service Data

Meteorological information is gathered from instrumentation operated by the IDEM Ambient Air Monitoring Section and the U.S. Weather Service, the National Park Service and various reporting agencies within the state. Parameters include: temperature, relative humidity, barometric pressure, rainfall, wind speed, and wind direction. No changes to the way in which these data are collected are anticipated due to the addition of the Fine Particulate data to the IDEM ambient air monitoring program.

## 19.0 Data Management

# 19.1 Background and Overview

This section describes the data management operations pertaining to PM<sub>2.5</sub> measurements for the SLAMS stations operated by the Indiana Department of Environmental Management. This includes an overview of the mathematical operations and analyses performed on raw PM<sub>2.5</sub> data. These operations include data recording, validation, transformation, transmittal, reduction, analysis, management, storage, and retrieval.

Data processing steps are integrated, to the extent possible, into the existing data processing system used for SLAMS network. Originally, all data was entered manually and was processed using a set of programs written by IDEM staff. Modems and data acquisition system (DAS) units now allow more immediate access to the site information. Loggers provide data collection for continuous analyzers at each station. Some sites use computers to monitor and record parameters such as sampler status, flow rate, and temperatures.

## 19.2 Data Recording

Data entry, validation, and verification functions are all integrated in the  $PM_{2.5}$  program. Procedures for filling out the laboratory sheets and subsequent data entry are provided in SOPs and are available on request.

#### 19.3 Data Validation

Data validation confirms that data processing operations have been carried out correctly. Once problems are identified, data is either corrected or invalidated. Error flags or QA status flags are saved as separate fields in the database so that it is possible to recover original data.

The following validation functions are incorporated into the  $PM_{2.5}$  databases to ensure quality of data entry and data processing operations:

• Range Checks - most parameters have programmed range checks. For example, valid times must be between 00:00 and 23:59. Staff recognize these ranges and notify proper personnel immediately when an entry is out of range.

- Completeness Checks Completeness criteria must be met when data is processed. For example, each filter must have a start time, an end time, an average flow rate, dates weighed, and operators' and technicians' names.
- Internal Consistency and Other Reasonableness Checks Several other internal consistency checks are used by the IDEM. For example, the end time for a filter must be greater than the start time. Computed filter volume (integrated flow) must be approximately equal to the exposure time multiplied by the nominal flow. Additional consistency and other checks are implemented when problems are encountered during data screening.
- Data Retention Raw data sheets are on file at the Shadeland facility for a minimum of three years. After three years have elapsed, hard copy records and computer backup media are cataloged and boxed for storage. Physical samples such as filters are discarded with appropriate attention to proper disposal of potentially hazardous materials.
- Statistical Data Checks Errors found during statistical screening are traced back to original data entry files and, if necessary, back to the raw data sheets. These checks are done on a monthly schedule and prior to any data submission to AQS.

Two key operational criteria for  $PM_{2.5}$  sampling are bias and precision. As defined in 40 CFR Part 58, Appendix A, these are based on differences between collocated sampler results and FRM performance evaluations. The IDEM inspects the results of collocated sampling during each batch validation activity. This data is evaluated as early in the process as possible so that potential operational problems can be addressed. The objective of the Air Monitoring Branch is to optimize the performance of its  $PM_{2.5}$  monitoring equipment.

#### 19.4 Data Transformation

The following relationships in Table 27 pertain to  $PM_{2.5}$  monitoring:

Table 27
Raw Data Calculations

Parameter	Unit	Type of Conversion	Equation
Filter Volume (V <sub>a</sub> )	m <sup>3</sup>	Calculated from average Flow Rate ( $Q_{avg}$ ) in l/min, and total elapsed time (t) in min. multiplied by the unit conversion ( $m^3/L$ )	$V_a = Q_{avg} * t * 10^3$
Mass on Filter (M <sub>2.5</sub> )	μg	Calculated from filter post-weight $(M_f)$ in mg and filter pre-weight $(M_i)$ in mg, multiplied by the unit conversion $(\mu g/mg)$	$PM_{2.5} = (M_f - M_i) * 10^3$
PM <sub>2.5</sub> Conc. (C <sub>PM2.5</sub> )	μg/m <sup>3</sup>	Calculated from laboratory data and sampler volume	$PM_{2.5} = M_{2.5}/V_a$

#### 19.5 Data Transmittal

Data transmittal occurs when data is transferred from one person or location to another or when data is copied from one form to another. Examples of data transmittal are: copying raw data from a notebook onto a data entry form for keying into a computer file and electronic transfer of data over a telephone or computer network.

The IDEM reports all PM<sub>2.5</sub> ambient air quality data and information specified by the AIRS Users Guide (Volume II, Air Quality Data Coding, and Volume III, Air Quality Data Storage). Such air quality data and information is screened and validated and then submitted directly to the AIRS-AQS via electronic transmission, in the AIRS-AQS format, and in accordance with the quarterly schedule. The specific quarterly reporting periods and due dates are shown in the Table 28.

Table 28
Data Reporting Schedule

Reporting Period	<b>Due Date</b>
January 1-March 31	June 30
April 1-June 30	September 30
July 1-September 30	December 31
October 1-December 31	March 31 (following year)

# 19.6 Data Reduction

Data reduction involves aggregating and summarizing results so that they can be understood and interpreted in different ways. The  $PM_{2.5}$  monitoring regulations require certain summary data to be computed and reported regularly to EPA. Other data is reduced and reported for other purposes such as station maintenance. Examples of data summaries include:

- average PM<sub>2.5</sub> concentration for a station or set of stations for a specific time period
- accuracy, bias, and precision statistics based on accumulated data
- data completeness reports based on numbers of valid samples collected during a specified period

The Audit Trail is another important concept associated with data transformations and reductions. An audit trail is a data structure that provides documentation for changes made to a data set during processing. Typical reasons for data changes that would be recorded include the following:

- corrections of data input due to human error
- application of revised calibration factors

- addition of new or supplementary data
- flagging of data as invalid or suspect
- logging of the date and times when automated data validation programs are run

Audit records include the following fields:

- operator's identity
- date and time of change
- table and field names for the changed data item
- reason for change
- information for changed item (date, time, site location, parameter, etc.)
- value before and after change

# 19.7 Data Tracking

The PM<sub>2.5</sub> program contains the necessary input functions and reports necessary to track and account for the location of filters and the status of data processing operations for specific data. Information about filter location is updated at distributed data entry terminals at the points of significant operations. The following input locations are used to track filter location and status:

# Laboratory

- 1. Filter receipt (by lot)
- 2. Filter pre-sampling weighing (individual filter number first enters the system)
- 3. Filter packaged for the laboratory (filter numbers in each package are recorded)

Shipping (package numbers are entered for both sending and receiving)

## Laboratory

- 1. Package receipt (package is opened and filter numbers are logged in)
- 2. Filter post-sampling weighing
- 3. Filter archival

## 19.8 Data Storage and Retrieval

Data archival policies for the  $PM_{2.5}$  data are shown in Table 29.

# Table 29 Data Archive Policies

Data Type	Medium	Location	Retention Time	<b>Final Disposition</b>
Weigh records; chain-of-custody forms	Hardcopy	Laboratory	3 years	Discarded
Lab Notebooks	Hardcopy	Laboratory	3 years	N/A
Field Notebooks	Hardcopy	Air Monitoring Branch	3 years	Discarded
PM <sub>2.5</sub> Database (excluding Audit Trail records)	Electronic (on-line)	Air Monitoring Branch	Indefinite (move to backup media after 5 years)	Backup tapes retained indefinitely
Audit Trail records	Electronic (backup tapes)	Air Quality Division	3 years	Discarded
Filters	Filters	Laboratory	3 years	Discarded

Security of data in the PM<sub>2.5</sub> database is ensured by the following controls:

- password protection on the database that defines three levels of access to the data
- regular password changes
- logging of all incoming communication sessions
- storage of media including backup tapes in locked, restricted access areas

# 20.0 Assessments and Response Actions

The results of quality assurance assessments indicate whether the control efforts are adequate or need to be improved. Documentation of all quality assurance and quality control efforts implemented during the data collection, analysis, and reporting phases is important to data users, who can then consider the impact of these control efforts on the data quality. Both qualitative and quantitative assessments of the effectiveness of these control efforts will identify those areas most likely to impact the data quality and to what extent.

Periodic assessments of SLAMS data quality are required to be reported to EPA. The selection and extent of the QA and QC activities used by a monitoring agency depend on a number of local factors such as the field and laboratory conditions, the objectives for monitoring, the level of the data quality needed, the expertise of assigned personnel, the cost of control procedures, pollutant concentration levels, etc.

In order to ensure the adequate performance of the quality system, the Indiana Department of Environmental Management performs the following assessments:

- Management Systems Reviews (MSR)
- Network Reviews
- Technical Systems Audits (TSA)
- Audits of Data Quality
- Data Quality Assessments (DQA)

## 20.1 Assessment Activities and Project Planning

# **20.1.1** Management Systems Review (MSR)

A Management Systems Review is a qualitative assessment of a data collection operation or organization to establish whether the prevailing quality management structure, policies, practices, and procedures are adequate for ensuring that the type and quality of data needed are obtained. Management systems reviews of the Ambient Air Monitoring Program are conducted every three years. The MSR will use appropriate federal regulations, and the QAPP to determine the adequate operation of the air program and its related quality system. The quality assurance activities for all criteria pollutants including PM<sub>2.5</sub> are part of the MSR. Office of Air Quality groups that are included in the MSR are the Quality Assurance, Ambient Monitoring, and Toxics Sections.

#### 20.2 Network Reviews

Conformance with network requirements of the Ambient Air Monitoring Network set forth in 40 CFR Part 50, Appendix L, CFR Part 58 Appendices D and E are determined through annual network reviews of the ambient air quality monitoring system. The network review is used to determine how well a particular air monitoring network is achieving its required air monitoring objective and how it should be modified to continue to meet its objective. A PM<sub>2.5</sub> network review is performed every year. Since the EPA is also required to perform these reviews, the IDEM coordinates its activity with Region 5 in order to perform the activity at the same time. The Quality Assurance Section is responsible for conducting the network review.

The following criteria are considered during the review:

- date of last review
- areas where attainment/nonattainment or re-designation
- results of special studies, saturation sampling, point source oriented ambient monitoring, etc.
- proposed network modifications since the last network review

In addition, pollutant-specific priorities may be considered (e.g., newly designated nonattainment areas, "problem areas", etc.).

Prior to the implementation of the network review, significant data and information pertaining to the review is compiled and evaluated. Such information will include the following:

- network files (including updated site information and site photographs)
- AQS reports
- air quality summaries for the past five years for the monitors in the network
- emissions trends reports for major metropolitan areas
- emission information, such as emission density maps for the region in which the monitor is located and emission maps showing the major sources of emissions

Discrepancies are noted on the checklist and resolved during the review. The following categories are emphasized during network reviews:

Number of Monitors - The number of SLAMS monitors required for PM<sub>2.5</sub> depends on the measurement objectives as discussed in 40 CFR Part 50, Appendix L, CFR Part 58 with additional details in the *Guidance for Network Design and Optimum Exposure for PM*<sub>2.5</sub> and  $PM_{10}$ . Adequacy of the network is determined by using the following information:

- maps of historical monitoring data
- maps of emission densities
- dispersion modeling
- special studies/saturation sampling
- best professional judgment
- SIP requirements
- revised monitoring strategies

Location of Monitors - For SLAMS, the location of monitors is not specified in the regulations, but is determined by the Regional Office and the Department of Environmental Management on a case-by-case basis to meet the monitoring objectives specified in 40 CFR Part 50, Appendix L and CFR Part 58, Appendix D. Adequacy of the location of monitors can only be determined on the basis of stated objectives. Maps, graphical overlays, and GIS-based information is helpful in visualizing or assessing the adequacy of monitor locations. Plots of potential emissions and/or historical monitoring data versus monitor locations will also be used.

During the network review, the stated objective for each monitoring location or site (Section 10) is confirmed and the spatial scale is verified and compared to each location to determine whether these objectives can still be attained at the present location.

Conformance to 40 CFR Part 50, Appendix L and CFR Part 58 Appendix E - Probe Siting Requirements - Applicable siting criteria for SLAMS are specified in 40 CFR Part 50, Appendix L and CFR 58, Appendix E, is assessed during an on-site visit by QA personnel. The on-site visit consists of physical measurements and observations to determine compliance with the Appendix E requirements. Since many of the parameters do not change within one year, this check at each site is performed every 3 years.

Prior to the site visit, the reviewer reviews the following:

- hard copy of the site description (including any photographs)
- data on the seasons with the greatest potential for high concentrations for specified pollutants
- seasonal predominant wind direction

A checklist similar to the EPA Region 5 form is used by state personnel. This checklist is found in the *SLAMS/NAMS/PAMS Network Review Guidance* which is intended to assist the reviewers in determining conformance with Appendix E. In addition to the items on the checklist, the reviewer performs the following tasks:

- ensure that the inlet is clean
- check equipment for missing parts, frayed cords, damage, etc.
- record findings in field notebook and/or checklist
- take photographs/videotape in the 8 directions (N, NW, W, SW, NE, E, SE, and S)
- document site conditions, with additional photographs/videotape

In addition to the items included in the checklists, other subjects for discussion as part of the network review and overall adequacy of the monitoring program include:

- installation of new monitors
- relocation of existing monitors
- siting criteria problems and suggested solutions
- problems with data submittal and data completeness
- maintenance and replacement of existing monitors and related equipment
- quality assurance problems
- air quality studies and special monitoring programs

A report of the network review must be written within two months of the review (Section 21) and appropriately filed (Section 10).

# **20.3** Technical Systems Audits (TSA)

A TSA is a thorough and systematic onsite qualitative audit, where facilities, equipment, personnel, training, procedures, and record keeping are examined for conformance to the QAPP. TSAs of the  $PM_{2.5}$  network should be accomplished every 3 years and will stagger the required TSA conducted by EPA Region 5. The QA Section will implement the TSA either as a team or as an individual auditor. The QA Office will perform 3 TSA activities that can be accomplished separately or combined:

- field-handling, sampling, shipping
- laboratory Pre-sampling weighing, shipping, receiving, post-sampling weighing, archiving, and associated QA/QC
- data management Information collection, flagging, data editing, security, upload

Key personnel to be interviewed during the audit are those individuals with responsibilities for: planning, field operations, laboratory operations, QA/QC, data management, and reporting.

To increase uniformity of the TSA, an audit checklist should be developed and used.

The audit team will prepare a brief written summary of findings, organized into the following areas: planning, field operations, laboratory operations, quality assurance/quality control, data management, and reporting. Problems with specific areas are discussed and an attempt made to rank them in order of their potential impact on data quality.

An audit report form is under development. This form is designed such that one is filled out for each major deficiency that requires formal corrective action. The finding should include items like: pollutant(s) impacted, estimated time period of deficiency, site(s) affected, and reason for action. The audit form will inform the appropriate section about serious problems that may compromise the quality of the data and therefore require specific corrective actions. They are initiated by Quality Assurance Section's project leaders, and discussed at a 'debriefing' with designated staff.

Post-Audit Activities - The major post-audit activity is the preparation of the systems audit report.

- audit title and number and any other identifying information
- audit team leaders, audit team participants, and audited participants
- background information about the project, purpose of the audit, dates of the audit, particular measurement phase or parameters that were audited, and a brief description of the audit process
- summary and conclusions of the audit and corrective action required
- attachments or appendices that include all audit evaluations and audit finding forms

To prepare the report, the audit team meets and compares observations with collected documents and results of interviews and discussions with key personnel. Expected QA Project Plan implementation is compared with observed accomplishments and deficiencies and the audit findings are reviewed in detail. Within thirty (30) calendar days of the completion of the audit, the audit report is prepared and submitted. The systems audit report is submitted to the appropriate branch managers and appropriately filed (Section 10).

If the Section Chiefs have written comments or questions concerning the audit report, the Audit Team will review and incorporate them as appropriate, and subsequently prepare and resubmit a

report in final form within thirty (30) days of receipt of the written comments. The report will include an agreed-upon schedule for corrective action implementation.

Follow-up and Corrective Action Requirements - The QA Section and the audited Sections work together to solve problems and to take the required corrective actions.

# **20.4** Audit of Data Quality (ADQ)

An ADQ reveals how the data is handled, what judgments were made, and whether all necessary corrections were made. ADQs can often identify the means to correct systematic data reduction errors. An ADQ is performed every year and will also be part of the TSA (every 3 years). Thus, sufficient time and effort is devoted to this activity so that the auditor or team has a clear understanding and complete documentation of data flow. Pertinent ADQ questions will appear on the TSA check sheets (currently being developed) to ensure that the data collected at each stage maintains its integrity. The ADQ will serve as an effective framework for organizing the extensive amount of information gathered during the audit of laboratory, field monitoring, and support functions within the agency. The ADQ follow the same reporting/corrective action procedures as the TSA.

# 20.5 Data Quality Assessments (DQA)

A Data Quality Assessment (DQA) is the statistical analysis of environmental data to determine whether the quality of data is adequate to support the decision which are based on the DQOs. Data is appropriate if the level of uncertainty in a decision based on the data is acceptable. The DQA process is described in detail in *Guidance for the Data Quality Assessment Process*, EPA QA/G-9 and is summarized below.

- 1. Review the Data Quality Objectives (DQOs) and sampling design of the program: review the DQO and develop one, if it has not already been done. Define statistical hypothesis, tolerance limits, and/or confidence intervals.
- 2. Conduct preliminary data review. Review Precision & Accuracy and other QA reports; calculate summary statistics, plots and graphs. Look for patterns, relationships, or anomalies.
- 3. Select the statistical test: Select the best test for analysis based on the preliminary review, and identify underlying assumptions about the data for that test.
- 4. Verify test assumptions: Decide whether the underlying assumptions made by the selected test hold true for the data and the consequences.
- 5. Statistical test: Perform test and document inferences. Evaluate performance for future use.

Data quality assessment is included in the *QA Annual Report*. Details of these reports are discussed in Section 21.

Measurement uncertainty is estimated for both automated and manual methods. Terminology associated with measurement uncertainty is found within 40 CFR Part 50, Appendix L and Part 58, Appendix A and includes: (a) Precision - a measurement of mutual agreement among individual measurements of the same property usually under prescribed similar conditions, expressed generally in terms of the standard deviation; (b) Accuracy- the degree of agreement between an observed value and an accepted reference value; accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; (c) Bias- the systematic or persistent distortion of a measurement process which causes errors in one direction. The individual results of these tests for each analyzer are reported to EPA.

Estimates of the data quality are calculated on the basis of single monitors and aggregated to all monitors.

# 20.6 Documentation of Assessments

Table 30 summarizes each of the assessments discussed above.

Table 30 Assessment Summary

Assessment Activity	Frequency	Personnel Responsible	First Scheduled	Report Completion	Reporting/Resolution
MSR	1/3 years	Air Monitoring	1/1/2000	30 days after	Air Management
N. I.D.	,	Branch Chief		activity	Assistant Comm.
Network Reviews					
Appendix D	1/ year	QA	1/1/2000	30 days after	Air Monitoring
Appendix E	1/3 years	Section Chief	1/1/2000	activity	Branch Chief
Technical	1/3 years	QA	5/1/99	30 days after	Air Monitoring
Systems Audits	175 years	Section	3/1/99	activity	Branch Chief
Audits of Data	1/ year	QA	5/1/99	30 days after	Air Monitoring
Quality	17 year	Section	3/1/99	activity	Branch Chief
Doto Quality		QA/Air		120 days	Air Monitoring
Data Quality Assessment	1/year	Monitoring	1/1/2000	after end of	Branch Chief/
Assessment		Sections		calendar yr	EPA Region

# 21.0 Reports to Management

This section describes the quality-related reports and communications to management necessary to support  $PM_{2.5}$  network operations and the associated data acquisition, validation, assessment, and reporting. Unless otherwise indicated, data pertaining to  $PM_{2.5}$  is included in reports containing monitoring data for other pollutants.

Important benefits of regular QA reports to management include the opportunity to alert the various Sections of data quality problems, to propose viable solutions to problems, and to procure necessary additional resources. Quality assessment, including the evaluation of the technical systems, the measurement of performance, and the assessment of data, is conducted to help ensure that measurement results meet program objectives and to ensure that necessary corrective actions are taken early, when they are most effective.

Effective communication among all personnel is an integral part of a quality system. Regular, planned quality reporting provides a means for tracking the following:

- adherence to scheduled delivery of data and reports
- documentation of deviations from approved QA and test plans, and the impact of these deviations on data quality
- analysis of the potential uncertainties in decisions based on the data

# 21.1 Frequency, Content, and Distribution of Reports

Required reports to management for PM<sub>2.5</sub> monitoring and the SLAMS program in general are discussed in various sections of 40 CFR Parts 50, 53, and 58. Guidance for management report format and content are provided in guidance developed by EPA's Quality Assurance Division (QAD) and the Office of Air Quality Planning and Standards (OAQPS). These reports are described in the following subsections.

## 21.2 QA Annual Report

Periodic assessments of SLAMS data quality is reported to EPA (40 CFR 58 Appendix A, Section 1.4, revised July 18, 1997). The IDEM's *QA Annual Report* is issued to meet this requirement. This document describes the quality objectives for measurement data and how those objectives have been met.

The *QA Annual Report* also provides for the review of the SLAMS air quality surveillance system on an annual basis to determine if the system meets the monitoring objectives defined in 40 CFR Part 58, Appendix D. Such review identifies needed modifications to the network such as termination or relocation of unnecessary stations or establishment of new stations which are necessary.

The *QA Annual Report* includes quality information for each ambient air pollutant in the IDEM's monitoring network. These sections are organized by ambient air pollutant category. Each section includes the following topics:

- program overview and update
- quality objectives for measurement data
- data quality assessment

For reporting PM<sub>2.5</sub> measurement uncertainties, the *QA Annual Report* contains the following summary information required by 40 CFR 58 Appendix A (Section 3.5, revised July 18, 1997):

- Flow Rate Audits (Section 3.5.1)
- Collocated Federal Reference Method Samplers (Section 3.5.2)
- Collocated Equivalent Samplers of same designation (Section 3.5.2)
- Assessment of Bias Using the FRM Audit Procedure (Section 3.5.3)

#### 21.3 Network Reviews

As required by 40 CFR Part 58 Appendix A, Section 4(a), revised July 18, 1997, the IDEM Ambient Monitoring Section Chief has provided a list of all monitoring sites and their AQS site identification codes to the EPA Region 5 office, with a copy to AQS. The Air Quality Subsystem (AQS) is EPA's computerized system for storing and reporting of information relating to ambient air quality data. If there is a change in this list of monitoring sites, the ambient air monitoring section chief will report this change to the EPA Region 5 office and to AQS.

# **21.4 Quarterly Reports**

Each quarter, the IDEM Quality Assurance Section will report to AQS the results of all precision, bias, and accuracy tests it has carried out during the quarter. The quarterly reports are submitted, consistent with the data reporting requirements specified for air quality data as set forth in 40 CFR Parts 58.26, 58.35 and 40 CFR Part 58 Appendix A, Section 4.

The data reporting requirements of 40 CFR Part 58.35 apply to those stations designated SLAMS or NAMS. Required accuracy and precision data are reported on the same schedule as quarterly monitoring data submittal. The required reporting periods and due dates are listed in Table 31.

Table 31 Quarterly Reporting Schedule

Reporting Period	Due on or Before
January 1-March 31	June 30
April 1-June 30	September 30
July 1-September 30	December 31
October 1-December 31	March 31 (following year)

In accordance with the Federal Register Notice of July 18, 1997, all QA/QC data collected is reported and is flagged appropriately. This data includes: "results from invalid tests, from tests carried out during a time period for which ambient data immediately prior or subsequent to the tests were invalidated for appropriate reasons and from tests of methods or analyzers not

approved for use in SLAMS monitoring networks . . . " (40 CFR Part 58 Appendix A, Section 4, revised July 18, 1997).

Air quality data submitted for each reporting period is edited, validated, and entered into the AIRS-AQS using the procedures described in the AIRS Users Guide, Volume II, Air Quality Data Coding. The Air Monitoring Branch is responsible for preparing the data reports, which are reviewed by the QA Section Chief before they are transmitted to EPA.

# 21.5 Technical System Audit Reports

The IDEM performs technical system audits of the monitoring system. The QA Section Chief issues these reports. Reports are filed and made available to EPA personnel during their technical systems audits.

External systems audits are conducted at least every 3 years by the EPA Regional Office as required by 40 CFR Part 58, Appendix A, Section 2.5. Further instructions are available from the EPA Regional QA Coordinator or the Systems Audit QA Coordinator, Office of Air Quality Planning and Standards, Emissions Monitoring and Analysis Division (MD-14), U.S. Environmental Protection Agency, Research Triangle Park, NC 27711.

# 21.6 Corrective Action Plans/Reports

Corrective action plans/reports are developed and implemented anytime problems are discovered by the different types of audits mentioned in these previous sections. Staff that may be included in the preparation of these corrective action plans/reports include:

- Office of Air Monitoring Branch Chief, who has the ultimate responsibility for the quality of the data and the technical operation of Indiana's PM<sub>2.5</sub> monitoring program
- Ambient Monitoring Section Chief, who is responsible for the operation of the network
- Quality Assurance Section Chief, who is responsible for establishing the QA policies and procedures employed by the Air Monitoring Branch
- Toxics Section Chief, who is responsible for the complete operation of the laboratory facility
- Project Leaders (for the 3 Sections mentioned above), who are responsible for the day-to-day operation of the network
- Field operators, technicians, analysts, and support staff, who physically carry out the PM<sub>2.5</sub> monitoring activities

# 22.0 Data Review, Validation, and Verification Requirements

This section describes how Indiana verifies and validates the data collection operations associated with the  $PM_{2.5}$  ambient air monitoring network. Verification is defined as confirmation by examination and provision of objective evidence that specified requirements have been fulfilled. Validation is defined as confirmation by examination and provision of

objective evidence that the particular requirements for a specific intended use are fulfilled.

Although there are a number of objectives for collecting ambient air data, the major objective for the IDEM PM<sub>2.5</sub> network is for comparison to the NAAQS and therefore, this is identified as the intended use. This section describes the verification and validation activities that occur at a number of the important data collection phases. Review and approval of this QAPP by the IDEM and the EPA provides initial agreement that the processes described in the QAPP, if implemented, will provide data of adequate quality. In order to verify and validate the phases of the data collection operation, the Department will use various qualitative assessments (i.e., technical systems audits, network reviews) to verify that the QAPP is being followed, and will rely on the various quality control samples, inserted at various phases of the data collection operation, to validate that the data will meet the DQOs.

# 22.1 Sampling Design

The objective of the sampling design is to represent the population of interest at adequate levels of spatial and temporal resolution. Most of these requirements have been described in the Code of Federal Regulations. However, it is the responsibility of the IDEM to ensure that the intent of the regulations are properly administered and carried out.

# 22.1.1 Sampling Design Verification

Verification of the sampling design will occur through three processes:

Network Design Plan Confirmation - The Network Design Plan that discusses the initial deployment of the network has been submitted, reviewed, and approved by EPA prior to implementation. This process verifies the initial sampling design.

Internal Network Reviews - Once a year, the Quality Assurance Section performs a network review to determine whether the network objectives, as described in the Network Design Plan, are still being met, and that the sites are meeting the CFR siting criteria (see Section 20).

External Network Reviews - Every 3 years, EPA Region 5 conducts a network review to determine whether the network objectives, as described in the Network Design Plan, are still being met, and that the sites are meeting the CFR siting criteria.

## 22.1.2 Sampling Design Validation

Ambient air data collected from network sites is used to validate the sampling design. Through the initial stages of implementation, Indiana used saturation monitors as well as special purpose monitors to validate that the monitors were properly sited and that the sampling design met the objectives of the network. This information is included in network review documentation and appropriately communicated to the EPA Region 5 office. In addition, the process described earlier is used to confirm the network design.

## **22.2 Sample Collection Procedures**

# 22.2.1 Sample Collection Verification

Sample collection procedures have been developed to ensure proper sampling and to maintain sample integrity. The following processes are used to verify the sampling collection activities:

Internal Technical Systems Audits - required every three years.

External Technical Systems Audits - conducted by the EPA Region 5 every three years.

Both types of technical systems audits are used to verify that the sample collection activity is performed as described in this QAPP and the SOPs. Deviations from the sample collection activity are noted and corrective action implemented.

# 22.2.2 Sample Collection Validation

The sample collection activity is just one phase of the measurement process. The use of QC samples that have been placed throughout the measurement process can help validate the activities occurring at each phase. The review of QC data such as the collocated sampling data, field blanks, the FRM performance evaluation, and the sampling equipment verification checks can be used to validate the data collection activities. Any data that indicates unacceptable levels of bias or precision is flagged and investigated.

# 22.3 Sample Handling

The following sections detail the requirements for sampling handling, including the types of sample containers and the preservation methods used to ensure that they are appropriate to the nature of the sample and the type of data generated from the sample. Due to the size of the filters and the nature of the collected particles, sample handling is one of the phases where inappropriate techniques can have a significant effect on sample integrity and data quality.

## 22.3.1 Verification of Sample Handling

Both internal and external technical systems audits are performed to ensure the specifications mentioned in the QAPP are followed. The audits include checks on the identity of the sample (i.e., proper labeling and chain-of-custody records), packaging in the field, and proper storage conditions (i.e., chain-of-custody and storage records) to ensure that the sample continues to be representative of its native environment as it moves through the data collection operation.

# 22.3.2 Validation of Sample Handling

Similar to the validation of sampling activities, the review of data from collocated sampling, field blanks, and the FRM performance evaluations can be used to validate the sample handling activities. Acceptable precision and bias in these samples would lead one to believe that the sample handling activities are adequate. Any data that indicates unacceptable levels of bias or precision is flagged and investigated.

# 22.4 Analytical Procedures

The following section details the requirements for the analytical methods, which include the presampling weighing activities that give each sample a unique identification, an initial weight, and prepares the sample for the field; and the post-sampling weighing activity, which provides the mass net weight and the final concentration calculations. The methods include acceptance criteria for important components of the procedures, along with suitable codes for characterizing each sample's deviation from the procedure

## 22.4.1 Verification of Analytical Procedures

Both internal and external technical systems audits are performed to ensure the analytical method specifications mentioned in the chapter are being followed. The audits will include checks on the identity of the sample. Deviations from the analytical procedures are noted and corrective action implemented.

## 22.4.2 Validation of Analytical Procedures

Similar to the validation of sampling activities, the review of data from lab blanks, calibration checks, laboratory duplicates, and other laboratory QC can be used to validate the analytical procedures. Acceptable precision and bias in these samples would lead one to believe that the analytical procedures are adequate. Any data that indicates unacceptable levels of bias or precision is flagged and investigated.

# 22.5 Quality Control

Sections of this QAPP specify the QC checks that are to be performed during sample collection, handling, and analysis. These include analyses of check standards, blanks, spikes, and replicates, which provide indications of the quality of data being produced by specified components of the measurement process. For each specified QC check, the procedure, acceptance criteria, and corrective action are specified.

## 22.5.1 Verification of Quality Control Procedures

Both internal and external technical systems audits are performed to ensure the quality control method specifications mentioned in the QAPP are followed.

# 22.5.2 Validation of Quality Control Procedures

Validation activities of many of the other data collection phases mentioned in this subsection use the quality control data to validate the proper and adequate implementation of that phase. Therefore, validation of QC procedures will require a review of the documentation of the corrective actions that were taken when QC samples failed to meet the acceptance criteria and the potential effect of the corrective actions on the validity of the routine data.

#### 22.6 Calibration

The following section, as well as the field and the analytical sections, details the calibration activities and requirements for the critical pieces of equipment for the  $PM_{2.5}$  network.

#### 22.6.1 Verification of Calibration Procedures

Both internal and external technical systems audits are performed to ensure the calibration specifications and corrective actions mentioned are followed. Deviations from the calibration procedures are noted and corrective action implemented.

#### 22.6.2 Validation of Calibration Procedures

Similar to the validation of sampling activities, the review of calibration data is used to validate calibration procedures. Calibration data within the acceptance requirements would lead one to believe that the sample collection measurement devices are operating properly. Any data that indicates unacceptable levels of bias or precision is flagged and investigated as described in previous sections. If this investigation leads to a discovery of inappropriate calibration procedures or equipment problems requiring corrective action, such action is implemented.

Validation includes the review of the documentation to ensure corrective action is taken as prescribed in the QAPP.

## 22.7 Data Reduction and Processing

# 22.7.1 Verification of Data Reduction and Processing Procedures

Both internal and external technical systems audits are performed to ensure the data reduction and processing activities mentioned in the QAPP are being followed.

# 22.7.2 Validation of Data Reduction and Processing Procedures

As part of the audits of data quality, a number of sample IDs, chosen at random, are identified. All raw data files, including the following are selected:

- pre-sampling weighing activity
- pre-sampling
- sampling (sampler download information)
- calibration the calibration information represented from that sampling period
- sample handling/custody
- post-sampling weighing
- corrective action
- data reduction

This data is reviewed and concentrations are checked to determine if the values in AQS compare to the data submitted. Data is reviewed to ensure that associated flags or any other data qualifiers have been appropriately associated with the data and that appropriate corrective actions are taken.

## 23.0 Validation and Verification Methods

Many of the processes for verifying and validating the measurement phases of the PM<sub>2.5</sub> data collection operation have been discussed in previous sections. If these processes, as written in the QAPP are followed, and the sites are representative of the boundary conditions for which they were selected, one would expect to achieve the PM<sub>2.5</sub> DQOs. However, exceptional events may occur during sampling, and field and laboratory activities may negatively affect the integrity of samples. In addition, it is expected that some of the QC checks will fail to meet the acceptance criteria. Information concerning problems that affect the integrity of data is identified in the form of flags. It is important to determine how these failures affect the routine data. The review of this routine data and their associated QC data is verified and validated on a sample batch basis. The sample batch is the most efficient entity for verification/validation activities. It is assumed that if measurement uncertainty can be controlled within acceptance criteria, at a batch level, then the overall measurement uncertainty is maintained within the precision and bias DQOs.

## 23.1 Process for Validating and Verifying Data

## 23.1.1 Verification of Sample Batches

After a sample batch is completed, a thorough review of the data is conducted for completeness and data entry accuracy. Data is reviewed for routine outliers and data outside of acceptance criteria. These data are flagged appropriately. All flagged data are "re-verified".

# 23.1.2 Validation

Validation of measurement data requires two stages, one at the measurement value level and the second at the batch level. Records of all invalid samples are filed. Information contained in the record includes a brief summary of why the sample was invalidated.

If the number of samples being invalidated is relatively small, the Quality Assurance Section reports them on a monthly basis to Region 5. If however, more than 5 values, in sequential order, from one site appear to require invalidation, Region 5 is notified and the issue described.

## 23.1.3 Validation of Measurement Values

Criteria are based upon CFR as well as field operator and laboratory technician judgment developed that are used to invalidate samples or measurements.

Example flags like those listed below may be used alone or in combination to invalidate samples.

Table 32
Single Flag Invalidation Criteria for Single Samples

Requirement	Flag	Comment	
Contamination	CON	Concurrence with lab staff and section chief	
Filter Damage	DAM	Concurrence with lab and field staff	
Event	EVT	Exceptional, known field event expected to have affected sample. Concurrence with lab and field staff	
Lab Accident	LAC	Concurrence with lab staff and section chief	
Field Accident	FAC	Concurrence with field staff and monitoring and quality assurance section chiefs	
Flow Rate Cutoff	FVL	Termination of sample collection due to flow rate > 10% design flow rate for 60 seconds.	

Table 33
Single Sample Validation

Requirement	Acceptance Criteria	Major	Minor	Flag
Flow Rate	≤ ±5% of 16.67 l/min for < 5 min	>10%	>5%	FLR
Flow Rate Verification	≤ 4% of transfer standard	> 6%	> 4%	FLV
Filter Temp	> 5 °C for < 30 min	> 10 °C	> 5 °C	FLT
Elapsed Sample Time	> 1380 or < 1500 minutes	Example: >1530 <1350	Example: >1440 and <1500 <1380 > 1350	EST
Holding Times Pre-sampling Sample Recovery	≤ 30 days ≤ 96 hours	>32 days >100 hours	>30 days >96 hours	HTE "
Post-sampling 25 °C 4 °C	≤ 10 days ≤ 30 days	>12 days >32 days	>10 days >30 days	"

## 24.0 Reconciliation with Data Quality Objectives

# 24.1 Reconciling Results with DQOs

The DQOs for the PM<sub>2.5</sub> ambient air monitoring network were developed in a previous section. The resulting DQOs are for precision, as measured by a coefficient of variation, to be less than 10% and for relative bias to be  $\pm 10\%$ . This section outlines the procedures that Indiana follows to determine if monitors and laboratory analyses are producing data that comply with the DQOs and what action is taken as a result of the assessment process. Such an assessment is termed a Data Quality Assessment (DQA) and is thoroughly described in *EPA QA/G-9: Guidance for Data Quality Assessment*.

# 24.1.1 Five Steps of DQA Process

As described in *EPA QA/G-9*, the DQA process is comprised of five steps.

Step 1. Review DQOs and Sampling Design. A previous section of this QAPP contains the details for the development of the DQOs, including defining the primary objective of the  $PM_{2.5}$  ambient air monitoring network ( $PM_{2.5}$  NAAQS comparison), translating the objective into a statistical hypothesis (3-year average of annual mean  $PM_{2.5}$  concentrations less than or equal to 15  $\mu$ g/m<sup>3</sup> and 3-year average of annual 98th

percentiles of the PM<sub>2.5</sub> concentrations less than or equal to 65  $\mu$ g/m<sup>3</sup>), and developing limits on the decision errors (incorrectly conclude area in non-attainment when it truly is in attainment no more than 5% of the time, and incorrectly conclude area in attainment when it truly is in non-attainment no more than 5% of the time).

Step 2. Conduct Preliminary Data Review. A preliminary data review is performed to uncover potential limitations to using the data, to reveal outliers, and generally to explore the basic structure of the data. The first step is to review the quality assurance reports. The second step is to calculate basic summary statistics, generate graphical presentations of the data, and review these summary statistics and graphs.

Review Quality Assurance Reports. The Air Monitoring Branch reviews all relevant quality assurance reports that describe the data collection and reporting process. Attention is directed to looking for anomalies in recorded data, missing values, and any deviations from standard operating procedures. This is a qualitative review; however, any concerns are further investigated in the next two steps.

Calculation of Summary Statistics and Generation of Graphical Presentations. Indiana generates some summary statistics for each of its primary and QA samplers. Summary statistics are calculated at the quarterly, annually, and three-year levels and include only valid samples. The summary statistics are: Number of samples, mean concentration, median concentration, standard deviation, coefficient of variation, and maximum and minimum concentrations.

These statistics are also calculated for the percent differences at the collocated sites. The results are summarized in a table. Particular attention is given to the impact on the statistics caused by the observations noted in the quality assurance review.

Step 3. Select the Statistical Test. The primary objective for the PM<sub>2.5</sub> mass monitoring is determining compliance with the PM<sub>2.5</sub> NAAQS. As a result, the null and alternative hypotheses are: where *X* is the three-year average PM<sub>2.5</sub> concentration and *Y* is the 3-year average of the annual 98th percentiles of the PM<sub>2.5</sub> concentrations recorded for an individual monitor. The null hypothesis is rejected; the area is not in compliance with the PM<sub>2.5</sub> NAAQS.

$$H_o = X \le 15 \,\mu g/m^3 \text{ and } Y \le 65 \mu g/m^3$$
  
 $H_A = X > 15 \,\mu g/m^3 \text{ and } Y > 65 \mu g/m^3$ 

Step 4. Verify Assumptions of Statistical Test. The assumptions behind the statistical test include those associated with the development of the DQOs in addition to the bias and precision assumptions. Their method of verification is addressed in this step. Note that when less than 3 years of data are available, this verification is based on as much data as are available.

Step 5. Draw Conclusions from the Data. Perform the calculations required for the statistical test and document the inferences drawn as a result of these calculations. If the design is to be used again, evaluate the performance of the sampling design.

The DQO is based on the annual arithmetic mean NAAQS. For each primary sampler, the IDEM determines if the PM<sub>2.5</sub> NAAQS was violated. In the DQO development, it is assumed that the annual standard is more restrictive than the 24-hour standard. If there are any samplers that violate ONLY the 24-hour NAAQS, then this assumption is not correct.

## 24.1.2 Action Plan Based on Conclusions from DQA

For this section, IDEM assumes that the previous variables used for developing the DQOs have been met. If this is not the case, Indiana must first revisit the impact of the violation on the bias and precision limits determined by the DQO process.

DQA indicates every monitor operated by Indiana is collecting  $PM_{2.5}$  mass data that is within the precision and bias goals determined by the  $PM_{2.5}$  DQOs.

If the conclusion from the DQA process is that each of the  $PM_{2.5}$  mass monitors are operating with less than 10% bias and 10% precision, then Indiana will pursue action to reduce the QA/QC burden. Possible courses of action include the following:

- Modifying the QA Monitoring Network. 40 CFR Part 50, Appendix L, CFR Part 58 requires that each QA monitor be the same designation as the primary monitor. Indiana is operating only R&P Partisol-Plus Air Samplers.
- Reducing QC Requirements. QC is integral to any ambient air monitoring network and is particularly important to new networks. However, once it is demonstrated that the data collected from the Indiana network are within tolerable levels of errors, a reduction in the QC checks such as those specified in Table 32 may be requested.
- Determine level of aggregation at which DQOs are violated. The DQA process can identify if monitors are having problems since the DQOs were developed at a monitor level. To determine corrective action levels, it must be determined whether the violation of the DQOs is due to problems unique to one or two sites, unique to Indiana, or caused by a broader problem, like a particular piece of equipment has poor QA on a national level. The AQS generates QA reports summarizing bias and precision statistics at the national and reporting organization levels, and by method designation. These reports will assist Indiana in determining the appropriate level at which the DQOs are being violated. The procedure for determining level of violation is:

- \* Review national reports for similar areas in which Indiana's DQA process indicates a violation. If large bias or imprecision is seen at the national level, Indiana will request assistance from the Region 5 and OAQPS.
- Communication with the Region 5 Office. If violations of the bias and precision DQOs are found, Indiana contacts the Regional 5 Office for assistance.
- Extensive Review of Quarterly Data until DQOs Achieved. Indiana continues to review quarterly QA reports and QC summaries until the bias and precision limits are attained.